

JPRS-JST-89-022

20 OCTOBER 1989



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# ***JPRS Report***

## **Science & Technology**

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***Japan***

JPRS-JST-89-022  
20 OCTOBER 1989

SCIENCE & TECHNOLOGY  
JAPAN

CONTENTS

AEROSPACE, CIVIL AVIATION

Space Environment Utilization Outlined [PUROMETEUSU, Feb 89].....	1
Space Station Project Described [PUROMETEUSU, Feb 89].....	4
FMPT Program Outlined [PUROMETEUSU, Feb 89].....	8
International Microgravity Laboratory [IML] Program [PUROMETEUSU, Feb 89].....	11
Free-Flying Unmanned Platform [PUROMETEUSU, Feb 89].....	14
Weightless Test Facility Proposed, Microgravity Experiments by Aircraft Reported [PUROMETEUSU, Feb 89].....	16
University Research on Space Environment Utilization [Ryo Horiuchi; PUROMETEUSU, Feb 89].....	20
Space Environment Utilization by Private Circles Discussed [PUROMETEUSU, Feb 89].....	24

## BIOTECHNOLOGY

Clinical Testing To Start on Stem Cell Growth Stimulator  
[JAPAN CHEMICAL WEEK, 8 Jun 89]..... 34

Biotechnology Research Themes Selected by MITI for FY 90  
[JAPAN CHEMICAL WEEK, 22 Jun 89]..... 35

## ENERGY

Energy Agency's 'Moonlight Project' Developments  
[Hidenobu Nakagawa; DENKI TO GASU, Feb 89]..... 36

## MICROELECTRONICS

NEC's SOR Lithography R&D Described  
[Koichi Okada; OPTRONICS, Mar 89]..... 49

## NUCLEAR ENGINEERING

Basic Research by Research Reactor  
[PUROMETEUSU, Dec 88]..... 59

Nuclear Fusion Reactor Core Research Status Examined  
[PUROMETEUSU, Feb 89]..... 66

## SUPERCONDUCTIVITY

Superconducting Oxide Fiber With Coating Film  
[S. Sakka; PROCEEDINGS OF NEW CARBON AND NEW CERAMICS  
FIBER CONFERENCE, 16 Dec 88]..... 75

Space Environment Utilization Outlined

43062506a Tokyo PUROMETEUSU in Japanese Feb 89 pp 16-18

[Article by Space International Section, Research and Development Bureau, Science and Technology Agency: "Feature Articles: Space Environment Utilization--Outline of Space Environment Utilization"]

[Excerpts] 2. Importance of Space Environment Utilization

Area of Material Science and Technology

First, space is expected to be useful for the advance of basic sciences because in space environments, especially in microgravitational environments, it is possible to observe phenomena difficult to observe or measure accurately on the ground, for example, mass transfer by Marangoni convection (convection due to surface tension) or diffusion.

Also, the aforementioned characteristics of space environments may be used for the manufacture of crystals without defects or composite alloys with uniform azimuths, the manufacture of composite materials with uniformly dispersed particles by the powder or melting method, the manufacture of high-purity materials by non-confinement flotation, the manufacture of other materials known to have excellent properties or functions but extremely difficult to produce on the ground and the creation of entirely new materials that have not yet been discovered.

Area of Life Science

In the area of life science, the utilization of space environments is expected to have the effect of explaining vital phenomena and drastically increasing the production of useful substances. For instance, the role of gravity in the evolution and development of life, such as cell division and differentiation, is an almost unknown area, and mutation using high-energy particles or radiation environments is expected to contribute to advances in life science.

Much is also expected of biotechnological applications, such as protein separation and monocrystal growth by electrophoresis using microgravity.

Although directed from a somewhat different point of view, research in space medicine is also an important theme in this area, and it must be studied promptly so that prolonged manned space flights can be realized. It is hoped that the expansion of manned space flights will be supported by the confirmation of physiological and psychological changes characteristic of space environments and the establishment of countermeasures against these changes. Also important are the selection of environmental conditions to assure human safety and the study of a closed ecological life support system.

#### Scientific Observation

Prolonged astronomical and space observation for extensive observation wavelength bands can be made possible by using space environments not affected by the earth's atmosphere. Therefore, scientific observations, including the observation of solar activities and the observation of gamma-rays, X-rays and gravity waves, are expected to progress.

#### 3. Japanese Response

As seen above, space environment utilization is effective for the creation of new materials, the promotion of life science, scientific observation and many other purposes, and will significantly affect not only the development of high technologies, but also socioeconomic advancement. Therefore, it is necessary that it be accelerated.

In Japan, the Science and Technology Agency and the National Space Development of Japan (NASDA) are preparing for the First Material Processing Test (FMPT) program, mainly on experimental themes concerning the creation of new materials, to be conducted by Japanese payload scientists, and are also preparing to participate in the International Microgravity Laboratory (IML) program and are proceeding with the Free Flyer program in cooperation with the Ministry of Education and the Ministry of International Trade and Industry. Furthermore, they are involved in the Space Station program, an international project, as will be stated later.

Private industries are also proceeding steadily with the utilization of space environments. They are testing it, using the Space Shuttle (GAS), small foreign rockets and the ballistic flights of aircraft.

Due to this tendency toward space environment utilization by private industry, the Space Environment Utilization Promotion Center (JSUP) was formed in February 1986 as a public service corporation under the joint control of the Science and Technology Agency and the Ministry of International Trade and Industry, and the Space Environment Utilization Research Institute was established in April 1986 as a company financed by the Japan Key Technology Center (Key Tech Center).

However, in, for example, the creation of new materials, space environments have disadvantages as well as advantages. Their problems include the difficulty of removing air bubbles or impurities, such as oxides, and the

fact that a temperature difference between the surface and the interior inevitably exists in materials exceeding a certain size. Therefore, in order to use them effectively, one must have abundant knowledge about the physical, chemical and biological phenomena that occur under weightlessness.

In order to use space environments on a regular basis, therefore, it is necessary to gather and analyze many data on phenomena under microgravity by experiments using aircraft and small rockets. From this point of view, the Science and Technology Agency has conducted "research on such subjects as the creation of new materials under weightless environments" (fiscal 1982-86) and "research to clarify physical, chemical and biological phenomena under microgravity through international cooperation" (Phase 1: fiscal 1987-89) as joint industrial-academic-governmental efforts financed from the Science and Technology Promotion Coordinating Fund. Also, the National Space Development Agency of Japan is developing space experiment common technologies with a view to using the Space Station.

#### 4. Prospects--Space Station Project

An agreement for space station cooperation was signed on 29 September 1988, outlining cooperation at various stages, beginning with the development of the space station, which will enable people to stay in space for a long time and drastically expand the capability of space activities by, among other things, increasing the power supply and working space.

There is no doubt that the use of the space station will advance and diversify into such areas as experiments on materials, life science, communications and engineering physics as well as scientific observation, and being able to use it freely is important to the future promotion of our science and technology and the improvement of our society.

In order to construct the space station, a wide range of areas of advanced science and technology must be developed and utilized, and this is believed to be extremely important when proceeding with Japan's future space development, including manned space activities. Furthermore, science and technology in general will progress and improve by developing areas of advanced science and technology, such as robotics, communications and materials, in addition to the achievements made by using space environments.

From this point of view, Japan will participate in the project with an experimental module of its own.

The Japanese development work will be conducted on a regular basis when the agreement on space station cooperation is ratified. In the space station, Japan will have its own space-utilization infrastructure, and its science and technology and national economy will greatly develop by making effective use of the space station.

Space Station Project Described

43062506b Tokyo PUROMETEUSU in Japanese Feb 89 pp 19-21

[Article by Space Utilization Promotion Subsection, Space Development Section, Research and Development Bureau, Science and Technology Agency: "On the Space Station Project"]

[Excerpt] 2. Outline of the Space Station

(1) The space station is composed of the multipurpose space station proper, to be constructed for a low earth orbit (LEO) at an altitude of about 460 km and an inclination of 28.5 degrees (Figure 1), and two polar-orbit platforms (POP) and a manned, tended Free Flyer (MTFF) to fly separately from the Space Station proper (Figure 2).

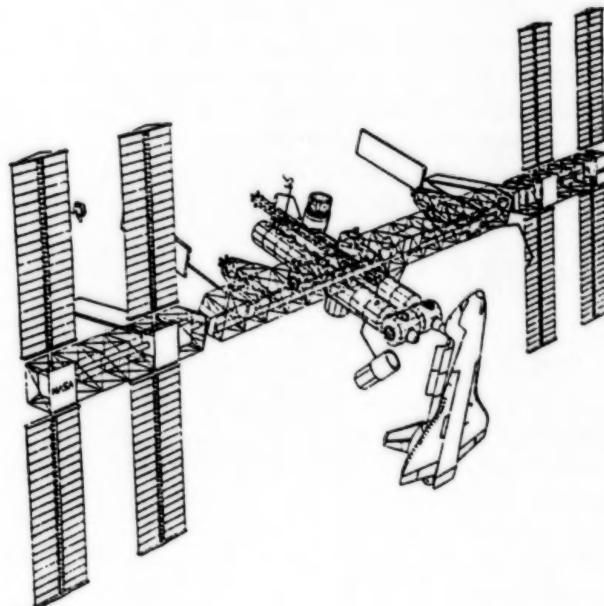


Figure 1. Space Station Proper

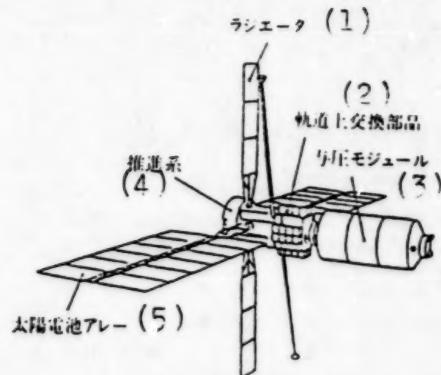


Figure 2. Manned, Tended Free Flyer (MTFF)

Key:

1. Radiator
2. Parts to be changed while in orbit
3. Pressurized module
4. Propulsive system
5. Solar cell array

The polar-orbit platforms will be provided by NASA and ESA. They have identical basic functions, can carry many pieces of observational equipment and surpass conventional satellites in cost efficiency by, among other things, being able to receive repair and fuel supply services by the space shuttle, and, therefore, are expected to be useful in the area of earth observation.

The manned, tended Free Flyer will be provided by ESA. It is equipped with an unmanned pressurized experiment module where automatic experiments on fluid mechanics, life sciences, material engineering, etc., will be conducted, taking advantage of its microgravity. It can be serviced periodically by being docked to the space station.

(2) The space station proper is a composite of elements provided by Japan, the United States, Europe and Canada, and is composed mainly of four cylindrical structures (modules) which are, as illustrated in Figure 1, configured near the center of gravity of the truss to obtain higher microgravity. Of the countries involved, the United States will develop and provide two modules, one for experimentation and the other for habitation, while Japan and Europe will each develop and provide one (for experimentation). As for Canada, it will develop and provide a mobile service complex (MSC) to be used for such purposes as assembling and maintaining the space station proper.

These components of the space station proper are fastened to a truss structure equipped with solar cells, radiation shields and propulsive systems to be provided by the United States.

(3) The space station proper will always be manned by a crew of eight, at the maximum, generally including one Japanese. The Japanese crew members will be selected and trained in Japan and the United States.

(4) The assumed life of the space station is more than 30 years and can gradually be prolonged.

### 3. Outline of the Japanese Experimental Module (JEM)

The Japanese experimental module (JEM) (Figure 6) is composed of a pressurized section, a supply section and an exposed section. The pressurized section is a multipurpose experimentation room which receives electricity, air, etc., since it is connected directly to the space station proper, permitting crew members to conduct experiments in an environment that does not require the use of special clothes, such as space suit, and which controls the exposed section and the manipulator. The exposed section is a facility exposed to space that handles changes of experimental devices, samples, etc., as well as the assembly of space structures via the manipulator and the air lock operated from the interior of the pressurized section. The supply section is the container used to supply, recover and transport samples, gases and machines necessary for the experiments and, in an emergency, functions to ensure the safety of crew members until they can be moved to a safe zone in the space station proper.

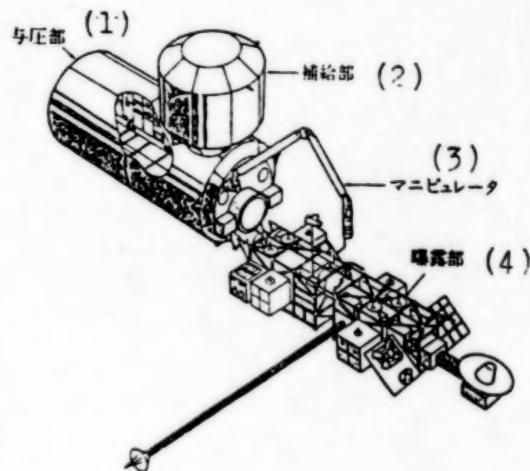


Figure 6. Experimental Module (JEM)

#### Key:

1. Pressurized section
2. Supply section
3. Manipulator
4. Exposed section

#### 4. Importance of Space Station

(1) Space environments, such as microgravity and high vacuum, can be utilized long and efficiently, with the construction of a space station.

(2) The space station can serve as the base for assembling large space structures or for repairing or supplying satellites.

For Japan, this project represents a very important opportunity to acquire science and technology concerning the use of space environments and manned space activities of which the Japanese accumulation of experience and technology has been minor.

What is more, the project will accelerate the space business of Japanese industry since it will make the regular utilization of space environments possible.

It is also important in its contribution to international society and in furthering cooperation with the countries in Europe and America.

**FMPT Program Outlined**

43062506c Tokyo PUROMETEUSU in Japanese Feb 89 pp 22-24

[Article by Space Experiment Group, National Space Development Agency of Japan: "FMPT"]

**[Excerpt] 3. Outline of FMPT [First Material Processing Test]**

FMPT is the first Japanese regular space experiment using the space shuttle to be manned by a Japanese PS (payload specialist). FMPT is also the beginning of Japanese development of manned support technology.

It proposes experimentation on 34 themes: 22 material experimental themes and 12 life science experimental themes.

The FMPT experiments will be conducted using about one-third of the space lab module (space experiment room) carried in the cargo bay of the space shuttle.

The FMPT flight program is tentatively scheduled as follows:

- \* Launching time: July 1991 (announced by NASA in August 1988)
- \* Launching/landing: Kennedy Space Center
- \* Flight period: about seven days (Experiments represent about 6.5 days of this period.)
- \* Orbit altitude: about 300 km, circular orbit
- \* Orbit inclination: 44 degrees
- \* Crew: seven  
One captain, one pilot, three mission specialists and two payload specialists (including a Japanese national)

**4. Selection of Experimental Themes**

In 1979, a Space Development Committee invitation was made soliciting public subscriptions for FMPT experimental themes. Then, after phased screening, 34 experimental themes covering many areas were finally selected in 1983.

These 34 themes were offered by 18 universities, 11 national research organizations and 5 other entities.

## 5. Outline of Experimental Themes

The 34 experimental themes can be classified as follows:

### (1) Material experiments (22 themes)

(a) Experiments to create new materials including experiments to manufacture compound semiconductors (11 themes)

(b) Experiments to improve existing ground production technologies, including experiments concerning metal manufacturing technologies (5 themes)

(c) Experiments to establish technologies basic to future space manufacturing, including studies concerning material manufacturing technologies, such as non-confinement process (6 themes)

### (2) Life science experiments (12 themes)

(a) Experiments to improve biological material separating and adjusting technologies, including experiments to separate biological components by electrophoresis (4 themes)

(b) Experiments to establish technologies for adaptation to space environments, including studies concerning the effects of space environments on organisms (8 themes)

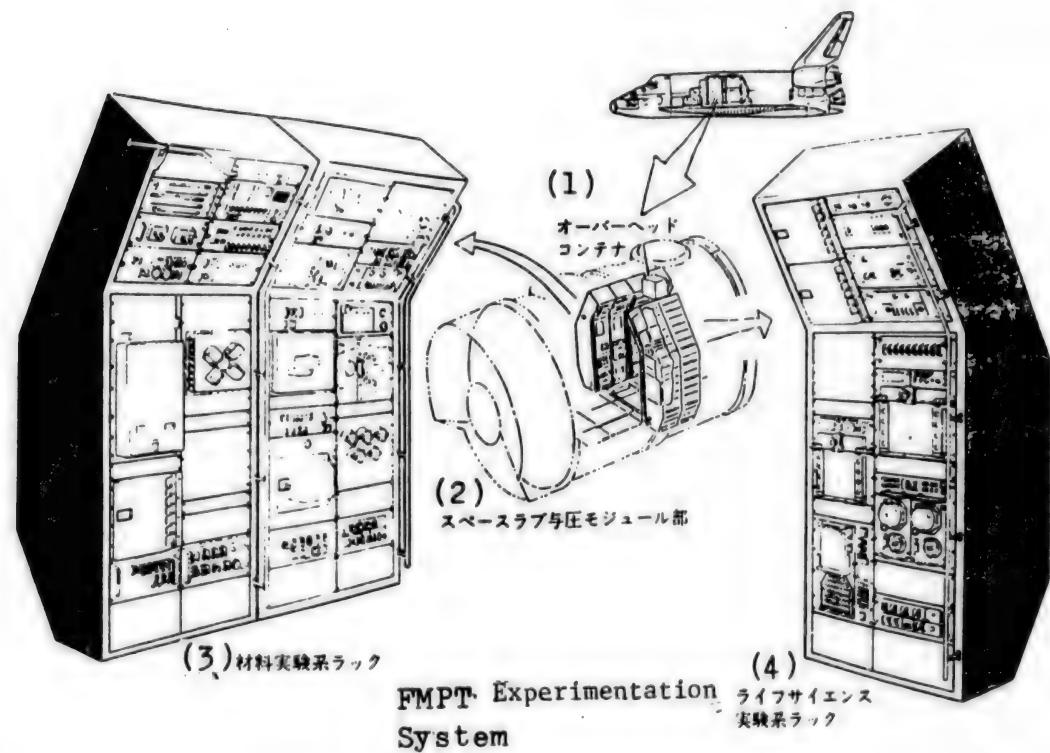
## 6. Development of Experimentation Equipment

The regular development of FMPT experimentation equipment was initiated in 1984 and was generally completed in March 1988 with the manufacture and testing of on board equipment.

The FMPT experimentation equipment comprises equipment and instruments for use in experiments involving the material and life science systems and is mounted on three double racks in the space lab module, as well as in such places as containers.

The experimentation equipment of the material experimenting system is composed of 10 units: 5 electric furnaces and 5 special experimentation devices.

The life science experimenting equipment is composed of six units, including electrophoretic devices.



**Key:**

1. Overhead container
2. Space lab pressurized module section
3. Racks for material experimentation system
4. Rack for life science experimentation system

**7. Japanese Payload Specialists (PS)**

Three Japanese payload specialist candidates were selected from among many applicants in 1985, and have since been undergoing training concerning experimental themes and experimentation equipment in preparation for space experiments.

Further training will take place in the United States for the year prior to launching.

**8. Conclusion**

FMPT is the first regular Japanese space experimentation project and is expected to succeed as an important pioneer project in the future era of space environment utilization.

International Microgravity Laboratory [IML] Program

43062506d Tokyo PUROMETEUSU in Japanese Feb 89 pp 24-27

[Article by Space Experiment Group, National Space Development Agency of Japan]

[Excerpts] 2. IML-1 Project

The launching to include the IML-1 project was first scheduled for 1987, but was later drastically changed following the Challenger accident.

The details of the experiments involved have already been established, and 201 researchers from 13 countries will experiment on 37 themes, using experimentation equipment provided by 5 countries: the United States, France, West Germany, Canada and Japan (Table 1). [not reproduced]

The two devices to be provided by Japan are small in scale, but are expected to produce significant results (Table 2).

Table 2. Outline of Experimental Themes to be Handled by Japan in IML-1

Experimental theme and outline	Experimentation device
o Crystal growth of organic superconductor utilizing weightless environment (Outline)	Organic crystal growth experimenter (OCGP) (with microgravity experimentation mechanism)
Large superior crystals of organic superconductor are developed, taking advantage of the weightless environment, and their physical properties are compared with those of crystals obtained on the ground.	
o Vibro-isolating effect by passive damping material (Outline)	
Vibro-isolating effect of passive damping material is tested with organic crystal growth experimenting device using passive damping material and similar device not using this material.	

[table continued on following page]

o Detection and analysis of high-energy cosmic rays by biological samples (Outline)      Radiation monitor control device (RMCD)

High-energy heavily-charged particles (H2E) infiltrating space ship are measured and their effects on biological samples (bacteria, animals, plants, etc.) are studied.

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### 3. IML-2 Project

The IML-2 project is now at the stage at which mission details are being studied, and the on board equipment to be used will be selected around March 1989.

Its experimental themes will be determined by the end of 1989. The payload experimenters will be selected by about October 1990, 24 months before launching. Japanese PSs may well be included.

A total of 56 experimentation devices have already been proposed by different countries (Table 3 [those of countries other than Japan not reproduced]). Japanese-proposed equipment will have to pass the forthcoming strict screening in order to be adopted as on-board items.

Since the Japanese-proposed equipment includes devices developed under the FMPT project and their improved versions, they have been noted by researchers from many countries and are fairly likely to be adopted. The experimental concepts incidental to the individual experimentation devices are based on the results of the FMPT project (Table 4).

Table 3. Japanese-Proposed Instruments for IML-2 Project

NASDA (Japan)
Temperature gradient electric furnace
Metal particle formation experimentation device
Organic crystal growth experimentation device
Protein/enzyme crystal device
Cell culture experimentation device/temperature and humidity support
Carrier-free electrophoretic device
Infrared telemeter

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Table 4. Japanese-Proposed IML-2 Candidate Experimentation Devices and Experimental Concepts

Experimentation device	Experimental concept
Temperature gradient electric furnace (GHF)	<ul style="list-style-type: none"> <li>o Preparation of alloys of oxide dispersion strengthening type</li> <li>o Explanation of solid-liquid interface stabilizing (destabilizing) factors under microgravity</li> <li>o Explanation for incorporating, excluding, separating and agglutinating behaviors of second liquid phase particles and gas bubbles on solid-liquid alloy interface</li> <li>o Study of mass transfer in melted matter</li> </ul>
Metal particle formation experimentation device (GEF)	<ul style="list-style-type: none"> <li>o Study of formation mechanism of gas-phase growth membrane</li> <li>o Experiment on diamond manufacturing by GEF device</li> <li>o Gas-phase growth of organic crystals</li> </ul>
Organic crystal growth experimentation device	<ul style="list-style-type: none"> <li>o On-the-spot observation of organic superconductor crystal growth in diffusion method</li> <li>o On-the-spot observation of solution growth of triglycin sulfade</li> </ul>
Carrier-free electro-phoretic device	<ul style="list-style-type: none"> <li>o Study of electrophoretic characteristics of granules with controlled charge in electrophoresis under microgravity</li> <li>o Separation of mammalian cells by free flow electrophoresis under microgravity</li> </ul>
Protein/enzyme crystal device	<ul style="list-style-type: none"> <li>o Experiment involving protein crystal growth in microgravitational environment</li> </ul>
Cell culture experimentation device/temperature and humidity support	<ul style="list-style-type: none"> <li>o Effect of microgravity on embryogeny of nematodes</li> <li>o Fission and multiplication of cultured mammalian cells in space</li> <li>o Inheritance and functional changes of monoclonal cells in space</li> <li>o Multiplication and development of cultured plant cells in space</li> <li>o Effect of microgravity on development and growth of plant callus</li> <li>o Adaptation of protozoans to microgravity</li> </ul>
Infrared telemeter	<ul style="list-style-type: none"> <li>o Fertilization and embryogeny of newt in space</li> <li>o Fertilization and embryogeny of sea urchin in space</li> </ul>

**Free-Flying Unmanned Platform**

43062506e Tokyo PUROMETEUSU in Japanese Feb 89 pp 27-30

[Article by Space Experiment Group, National Space Development Agency of Japan: "Free-Flying Unmanned Platform"]

**[Excerpts] 1. Outline**

The Free-Flying Unmanned Platform (Free Flyer) is a system designed to conduct unmanned space experiments for several months to a year according to different test requirements, taking advantage of microgravity, radiation environment, etc., after being released into an earth-circling orbit, about 300 km in altitude, by the space shuttle or some other vehicle.

It supposedly will offer the best microgravitational environment for a certain period because it is unmanned.

Chief among the past examples and future plans are the following:

**① Space Experiment/Observation Free Flyer (Space Flyer Unit) [SFU]**

SFU is the Free Flyer being developed jointly by the Institute of Space and Astronautical Science, the Ministry of International Trade and Industry and the Science and Technology Agency/National Space Development Agency of Japan (NASDA). The plan proposes that it be launched in winter 1993 by the H-II rocket now being developed by NASDA, and for it to be recovered by the space shuttle about 3 months later. Its system data include a diameter of 4.5 m, a height of 2.8 m, a length of about 20 m with stretched-out paddles, a weight of 3.5 tons and a power supply of 1.4 kW (Figure 2), while its functions include data down link, data storage, pointing and orbit change. The experiments to be conducted will cover many areas including science/engineering, microgravity and life science and the three government offices concerned will proceed with the study of these applications in parallel with system development (Table 3). Much is expected of the results of this space experiment, which is deemed important as a preliminary step to the regular operation of the space station.

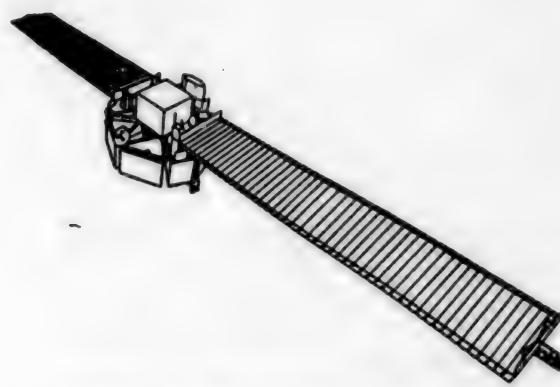


Figure 2. Configuration of SFU

Table 3. List of Experimentation Devices To Be Carried by SFU

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On-board experimenting device	Two-dimensional high voltage array experiment (2D/HVSA)
	Electric propulsion experiment (EPEX)
	Environment measuring experiment (EDP)
	Material experiment (MEX)
	Space geological experiment (BIO)
	Space infrared telescope (IRTS)
	JEM exposed section, partial model experimental device (EFFU)
	Composite heating furnace (CHF)
	Focal heating furnace (MHF)
	Singular heating furnace (IHF)

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Weightless Test Facility Proposed, Microgravity Experiments by Aircraft Reported  
43062506f Tokyo PUROMETEUSU in Japanese Feb 89 pp 31-33

[Article by Space Plan Section, Research and Development Bureau, Science and Technology Agency: "On Provision of Gravitational Drop Experimental Facility"]

[Excerpts] Means of Ground Experimenting Involving Weightlessness

Commercial service to realize weightlessness, using small rockets, is already in practice in West Germany and Sweden. In Japan, the National Space Development Agency conducted material and other experiments using weightlessness in 1980-1983 by launching small TT-500A rockets from the island of Tanegashima.

Japan has facilities for drop experiments at the National Aerospace Laboratories and Nagoya University, etc., but these are very small, with drop distances of about 10 m, and allow weightlessness to be maintained for only about a second. Therefore, Japanese researchers studying weightlessness strongly desire the provision of large drop experimental facilities with long drop distances in order to obtain longer durations of weightlessness.

Outline of Weightless Drop Experimental Facility Conceived by Gifu Prefecture

Gifu Prefecture has proposed a plan to provide an underground weightless drop experimental facility, free from the effects of meteorology, taking advantage of a shaft (depth: 150 m) in Toki City, Gifu Prefecture, in a uranium mine owned by the Power Reactor and Nuclear Fuel Development Corporation under the jurisdiction of the Science and Technology Agency.

The outline of this Gifu Prefecture-proposed weightless drop experimental facility is as follows:

(1) Site

Chubu Works, Power Reactor and Nuclear Fuel Development Corporation, Toki City, Gifu Prefecture (About 1 hour from Nagoya by electric train; vicinity of Toki Interchange, Chuo Expressway)

(2) Specifications of weightless drop experimental facility

1. Duration of weightlessness: 4.5 seconds (100-m free fall)
2. Quality of microgravity:  $10^{-4} \sim 10^{-5}$  G

3. Inside diameter of capsule: 80 cm

(3) Form of operation

1. Management: Semi-public corporation jointly financed by Gifu Prefecture, Toki City, etc.

2. Number of experiments conducted daily: About 10 at the most

3. Rates: Now being studied

(4) Construction schedule, etc.

Establishment of semi-public corporation: During first half of fiscal 1989

Start of construction: Middle of fiscal 1989

Start of operation: Probably end of fiscal 1990

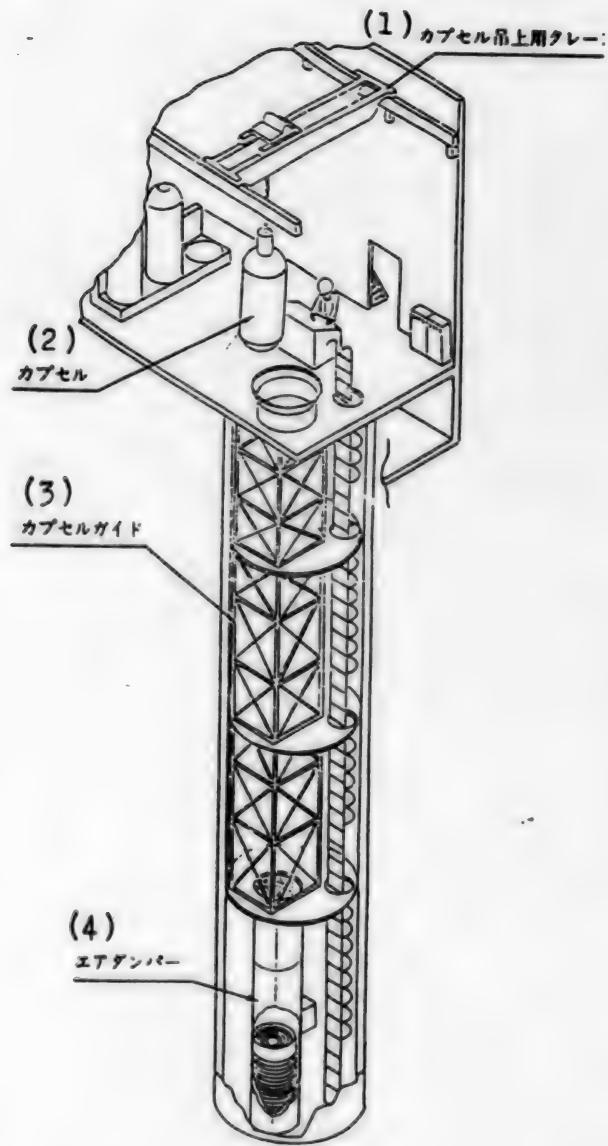
Principle of Provision of Weightless Drop Experimenting Facility

Gifu Prefecture wants to provide the weightless drop experimental facility in accordance with the recently enacted Multipolar Dispersion National Land Formation Expediting Law. The principle of this law is that if a development keypoint area basic concept prepared by a prefecture is approved by the competent minister under this law, facilities central to the concept may receive preferential taxational and financial treatment from the government.

Gifu Prefecture incorporates the weightless drop experimental facility of Toki City as central to its Tono Seibu Research and Academic City Plan comprising Toki and two other cities and a town, and has requested the Ministry of Finance, through the Science and Technology Agency, to receive interest-free loans from profits accrued from the sale of NTT shares in accordance with the Multipolar Dispersion National Land Formation Expediting Law. The fiscal 1989 request comprises 400 million yen from the NTT interest-free loans and 100 million yen from Japan Development Bank low-interest loans.

Use of Weightless Drop Experimental Facility

The prospective users of the weightless drop experimental facility include local ceramic enterprises, aerospace enterprises located in the Chubu Region and universities and national experimental and research institutions located throughout Japan.



Structure of Weightless Drop Experimental Facility

Key:

1. Capsule lifting crane
2. Capsule
3. Capsule guide
4. Air damper

## Microgravity Experiments by Aircraft--Science and Technology Promotion Coordinating Expenses

Since fiscal 1987, the Science and Technology Agency has conducted a "study concerning the elucidation of physical, chemical and biological phenomena under microgravitational environments through international cooperation" as a joint industrial-academic-governmental effort financed by science and technology promotion coordinating expenses.

As part of this study, experiments using microgravitational environments obtained by the ballistic flight of aircraft (MU-300) were conducted in November and December 1988. In microgravity experiments using aircraft, a microgravitational environment lasting about 20 seconds can be created by reducing the thrust of the aircraft while climbing to the extent of offsetting air resistance and causing it to fly parabolically (ballistically). The desired data are obtained through concurrent experimentation.

This study is being participated in by many national research organizations, universities and private enterprises, and is being conducted in cooperation with the United States and West Germany, etc. The recent aircraft experiments comprised a "study concerning mass and heat transfer in melted glass solution" by the Government Industrial Research Institute, Osaka, a "study concerning composite Malangoni convection" by the Ishikawajima-Harima Heavy Industries and a "study concerning the function to integrate the sense of equilibrium" by Nagoya University.

In the "study concerning mass and heat transfer in melted glass solution," which was intended to observe and analyze the form change and interfacial phenomena of melted glass solution under a microgravitational environment, researchers visiting from the Rolla campus of the University of Missouri, U.S.A., a partner in the joint research, conducted experiments using Japanese equipment.

The "study concerning composite Malangoni convection" consisted of basic experiments on equipment to photograph fluid data with high precision and the analysis of effects of concentration differences in Malangoni convection (convection due to differences in surface tension).

In the "study concerning the function to integrate the sense of equilibrium," the attitude control of the carp and the activities of its cerebellum under a microgravitational environment, as well as man's head/eyeball response and autonomic system responses (heart beat, respiration, etc.) under the same environment, were analyzed to explain space sickness.

**University Research on Space Environment Utilization**

43062506g Tokyo PUROMETEUSU in Japanese Feb 89 pp 40-43

**[Article by Ryo Horiuchi, professor, Institute of Space and Astronautical Science]**

**[Excerpt] Setup for Space Utilization Research**

In proceeding with the new area of space science generally known as space utilization, the Institute of Space and Astronautical Science created a Space Utilization Research Committee in 1987 to deliberate on the conduct of space utilization research at universities, etc. At the same time, a Space Station Utilization Research Center, to execute the decisions of the Research Committee, was added as an annex to the Institute of Space and Astronautical Science. In addition, many university and other researchers are conducting research on space environment utilization at their respective organizations and jointly with the Institute of Space and Astronautical Science. The preparation of this research setup is the direct result of advances in the space station project and has been initiated according to the report of the Space Station Special Subcommittee, Space Development Committee, requiring that the Institute of Space and Astronautical Science consolidate the station utilization requests of university and other researchers. However, the deliberations of the Space Utilization Research Committee are not limited to the space station, but encompass the mounting of experimental equipment on the Free Flyer (SFU), etc., and, of course, include basic ground experiments concerning space utilization as important activities.

The Space Utilization Research Committee is composed of researchers belonging to the Institute of Space and Astronautical Science and other universities. It deliberates while maintaining close contact with universities and other research organizations and researchers conducting advanced activities in the respective areas of space utilization research, and should be regarded by universities as the science board for space environment.

The Space Utilization Research Committee includes the International Response Group as a subcommittee because the space station project is being promoted through international cooperation. The International Forum on Scientific

Uses of Space Station (IFSUSS) is an organization which makes proposals to the project authority from the standpoint of international discussion and scientific utilization by scientists involved in the scientific uses of the space station. Japan is represented in it by this international response group known as the Japanese Science Advisory Group (JSAG).

The composition of the research team in the Space Utilization Research Committee may be presented as an example to show the areas and details of space environment utilization research at universities. Six central areas of space utilization research have been designated with each of these areas having its own research team. The six areas include astrophysics, solar system science, life science, earth observation, microgravity science and engineering physics experimentation. The three areas of astrophysics, solar system science and earth observation were established previously, and the scientific significance and maturity of tasks in these areas have already been evaluated. The necessity to develop microgravity science and life science as areas which would include the organization of researcher communities has been pointed out in the space station project interim report of the Space Science Section, Specific Research Area Promotion Division, Science Council. The three areas consisting of these two and engineering physics experimentation are areas where basic research must especially be promoted to advance the scientific significance of tasks and the maturity of experiment proposals. Partly for this purpose, these research teams have subteams to handle research contacts, thereby expediting the organizing of research. The composition of the subteams is within the framework of space utilization research designated by the Space Utilization Committee and specifically indicates the details of each area.

The subteams for microgravity science are as follows: The coagulation and crystal growth subteam mainly engages in explaining the effects of a micro-gravitational environment on phenomena related to the coagulation and crystal growth of metal materials and electronic functional materials. The fluid, heat transfer and basic process subteam studies the phenomenon of fluid heat transfer and gravity from a more basic point of view. Then, there is the gas phase and high vacuum utilization subteam for studying the use of the vast vacuum expanses of space for scientific experiments and the production of functional materials.

For life science, there are four subteams: outerspherical biology, animal system, plants/agronomy and medicine. Outerspherical biology covers from chemical evolution to the birth and evolution of life in space. The animal system aims to explain the basic principle of life by studying how space environments, such as microgravity and space radiation, affect cell-level and animal physiology and development included in basic biology. Plants/agronomy aims to elucidate how the cytoarchitecture and functional differentiation of plants and their morphological control are related to gravity, and to synthesize man's life environments in space. Medicine consists of basic research concerning the adaptation of the human body to space environments and its readaptation after returning to the earth.

The engineering physics experimentation research group is composed of four subteams: energy, propulsion, control/communications and heat/structure, with each subteam handling important elements in future space development.

It is urgent for the new area of space utilization research that the scientific significance of tasks be explained through basic research, advanced research concerning space experiment equipment and experimenting technologies and that this area be further developed. The Space Utilization Research Committee has been conducting these studies since fiscal 1988 using funds provided for space station utilization basic experiments. These basic research funds are allocated to experimental tasks publicly solicited from academic and other researchers according to rates determined after screening by the evaluation subcommittee, which is established by the research committee. In the initial fiscal year, public invitation was limited to three areas: microgravity science, life science and engineering physics. Of the 114 applications received, 13 tasks were adopted. These basic experimental funds are used to conduct preliminary ground experiments, evaluate their results and conduct more experiments on selected tasks using balloons and rockets. The development of large equipment and on-board instruments for use in the preliminary experiments is either the responsibility of the Institute of Space and Astronautical Science or is conducted with its technical assistance. The results will not only be announced at related scientific meetings, but will also be published in connection with use symposiums sponsored jointly by the Institute of Space and Astronautical Science and the Science Council of Japan or by publishing reports of results, and will be available for discussions by related researchers. The development of space experiment instruments deemed scientifically useful during this phased experimental application and evaluation will be initiated if they are to be carried on board the space station or the SFU. As for what merits use on the space station, the current procedure is for the Institute of Space and Astronautical Science to compile the requests for instrument use and forward them to the Space Development Committee.

#### Use of Space Environments and Future of Space Science

In light of space environment utilization becoming a mainstay of space activities during the 1990s, the furtherance of basic research at universities is important to the substantive use of space environments. The aforementioned research setup at universities and procedure of mounting for space experiments may seem to be somewhat behind the schedule of the space station project schedule. However, it is also a fact that planning, with emphasis on the domestically motivated development of science itself, assures the future rewards of space environment utilization. Particularly during the initial period of space environment utilization, academic research is more important than the development of practical use. It is necessary to accumulate and systematize knowledge by studying on the ground and in space to explain basic processes. Using space environments is important to the very progress of science. If learning, which has taken the earth's environment for granted, can reassess phenomena from the point of view of space and be restructured

as a more universal system of knowledge, it probably will be counted as an achievement of space development. Several attempts at scientific experimentation using the vast expanses of space as a laboratory have already been initiated and are succeeding. Perhaps, the 21st century can only be ushered in as a very fruitful century if the swell of the tidal current, i.e., space development, coincides with the advance of space science riding the crest of this wave.

## AEROSPACE, CIVIL AVIATION

### Space Environment Utilization by Private Circles Discussed

43062506h Tokyo PUROMETEUSU in Japanese Feb 89 pp 43-50

[Excerpts] [Introduction] The Japan Space Environment Utilization Promotion Center was established in February 1986 to expedite space environment utilization by the industry and has since handled such activities as surveys and research concerning the use of space environments. In addition, various surveys, research and development are also being conducted by individual private companies. Here, we shall examine the cases of the Society of Japanese Aerospace Companies (corporate entity) and the Space Environment Utilization Research Institute (corporation) with respect to space environment-using experimental projects being promoted by non-governmental-public organizations.

Part 1. Space Environment Utilization Experiments by GAS and Small Rockets  
[Section contribution by Akio Ajimine, chief, Space Division, Technical Department, Society of Japanese Aerospace Companies]

#### 1. Introduction

The Society of Japanese Aerospace Companies (incorporation entity) began in 1952 as Aviation Discussion Group and absorbed the work of the space branch of the Japan Rocket Development Council in 1974. It comprises regular members: Japanese companies manufacturing or repairing airframes, engines, artificial satellites, rockets, related equipment and materials (presently 145 companies), and associate members: trading firms, research institutes, etc., concerned with the aerospace business (presently 33 companies). It is the single private nonprofit industrial organization whose purpose is to aid in the sound development of the Japanese aerospace industry.

This society has promoted projects involving experimental use of space environments since fiscal 1985, focusing on GAS (Get Away Special), a simple means of experimentation using surplus space in the cargo bay of NASA's space shuttle, but the experiments had to be abandoned following the space shuttle accident. Therefore, in fiscal 1987 it began to proceed with the use of MASER, a small rocket of the Swedish Space Corporation (SSC), as a means certain to assure test opportunities and produce superior micro-gravitational environments at relatively low cost. These are thought to be useful as preliminary means of experimentation prior to regular space environment experiments using the proposed Space Station and Free Flyer, etc.

## GAS-Using Experiments

### (1) Gas Experiment System

Officially, GAS is termed a small-scale experiment payload. It consists of experimental equipment and materials, measuring instruments and a power source housed in a cylindrical aluminum container, about 50 cm in diameter, about 70 cm in height, which is surrounded by a heat-insulating material. Figure 1 [not reproduced] shows an example of its structure and Figure 2 shows it as it is attached to the sidewall of the space shuttle's cargo bay.

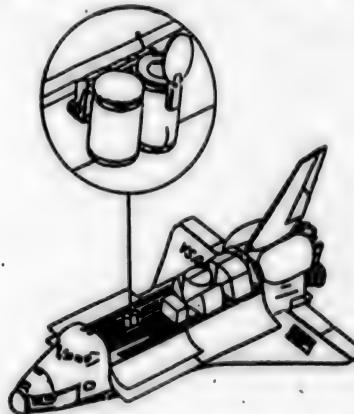


Figure 2. GAS as Mounted on Space Shuttle

### (2) Experimental Themes and Equipment

Twelve experimental themes relative to electronic materials and four biology-related themes were selected under the guidance of the Ministry of International Trade and Industry and with the cooperation of experts in this area. Their details are shown in Tables 1 and 2.

Table 1. GAS Experimental Themes Related to Electronic Materials

No.	Theme	Details	Remarks
1	Solution growth of GaAs single crystals	Describing growth speed and growth mechanism of GaAs crystals. (State of homogeneous formation by nonconvection)  Temperature is raised as Ga is surrounded by GaAs single crystal substrates.  Temperature is lowered after melting GaAs surface.	Sealed pipe heating 900°C

[Table continued on following page]

2	Solution growth of GaAs mixed crystals	Describing growth speed and growth mechanism of GaAs mixed crystals. (State of homogeneous formation by nonconvection)  For example, temperature is raised as Ga+As is surrounded by GaSb single crystals. Temperature is lowered after melting GaSb surface.	Sealed pipe heating 900°C
3	Heavy element doping to GaAs crystals	Comprehending state involving doping of heavy elements (Bi, etc.) to GaAs crystals. (Possibility of formation of mixed crystals by excessive doping)	Sealed pipe heating 900°C
4	Formation of InSbBi mixed crystal structure	Comprehending state involving doping of heavy elements (Bi, etc.) to InSb. (Formation of high-quality mixed crystal structure by increasing dosage of Bi dope)  Melting and coagulation of samples with some element ratios.	Temperature gradient furnace 650°C
5	Gas-phase growth of InGaAs	Describing gas-phase growth mechanism of compound semiconductor mixed crystals.  Growth of InGaAs is caused by providing temperature gradient in sealed pipe.	Temperature gradient furnace 800°C
6	Formation of GaAs/Ge heteroeutectoid	Determining prospects of obtaining super-lattice structure with same crystal-axis direction, using single crystals, by heterogeneous alloy eutectoid structure formation.  Melt temperature decrease in eutectoid section.  Directional coagulation.	Sealed pipe heating 900°C
7	Formation of compound semiconductor film single crystals on insulating substrate	Determining possibility of growth of compound semiconductor single crystals on insulating substrate.  Formation of InSb polycrystal membrane on quartz substrate. Melting recrystallizing film by movable strip heater.	500°C

[Table continues on following page]

8	Growth of crystals for optoelectronics use by diffusion process	<p>Acquisition of data for manufacturing high-quality crystals for optoelectronics use from saturated solution by diffusion process.</p> <p>For example, saturated solution of potassium hydrogenphosphate (KDP) and alcohol are separated in two chambers.</p> <p>Crystal formation by diffusion is caused by opening wall between chambers and thus, growth process is observed.</p>	Two-chamber solution mixing Video recording
9	Melting and coagulation of Si-Pb system	Determine whether Si-Pb system which conducts two-phase separation on ground produces homogeneous diffusion system under condition of weightlessness through melting coagulation experiments.	Sealed pipe heating 1450°C
10	Crystal growth of $\text{NbSe}_3$	<p>Acquisition of data on growth of one-dimensional electrically conductive materials from gaseous phase.</p> <p><math>\text{NbSe}_3</math> growth status is checked by providing temperature gradient in sealed pipe.</p>	Temperature gradient furnace 900°C
11	Sealed-pipe heating of NdFeB magnet material	Acquisition of data to explain coercive force generating mechanism of NdFeB system which comprises extremely strong magnet material. Examine magnetic characteristic, composition change and status of magnetic grain boundary.	Sealed pipe heating 1400°C
12	Observation of boiling by electric heating in medium	<p>Acquisition of data for developing heat storing and transfer equipment in space.</p> <p>Whether boiling causes air bubbles is examined by electric heating, with heater placed in water or some other medium.</p>	Video recording 36°C (Local heating 86°C)

Table 2. GAS Experimental Themes Related to Biology

No.	Theme	Details	Remarks
1	Experiment on electrophoresis	Several mixed enzymes are separated by electrophoresis, taking advantage of microgravity in space, and effects of separation are compared with those on ground.	Record: Visual data (VTR), temperature data, etc. Control: pump, power source, etc.
2	Bubble experiment	Gas/liquid separation experiment is conducted under microgravity environment as basic research necessary for developing element technology of aeration culture of yeasts under microgravitational environment, and method, conditions, etc., thereof are explained.	Record: picture data (VTR), temperature data, etc. Control: Pump, stirring, etc.
3	Tissue culture experiment	Cellular slime mold is cultured under microgravitational environment, status of its morphogenesis is recorded, and effects of gravity on structure at cell molecular level are studied.	Record: Visual data (35mm camera), temperature data, etc. Control: pump, electromagnetic valve, temperature, etc.
4.	Experiment on crystal growth of oxygen protein	Experiment involving growth of protein crystals is conducted under microgravitational environment and crystal growth process is recorded and analyzed.  Effectiveness of microgravitational environment as it affects growth of protein crystals is evaluated.	Record: Visual data (35mm camera), temperature data, etc. Control: mirror, temperature, etc.

Developing the experimental apparatus was the responsibility of the Mechanical System Promotion Association, which promoted the development by creating a committee composed of experts. In the area of electronic materials, the 12 themes were divided into 3 experimental groups, with NEC Corporation in charge of manufacturing the 3 experimental systems. In the biology-related area, Fujitsu Ltd. took charge of manufacturing four experimental systems catering to the respective themes.

The launching of the space shuttle was resumed in September this year after a 32-month suspension, and we hope that the delayed GAS experiment will be begun as soon as possible.

### Experiments Using Small Rockets

#### (2) Experimental Themes and Equipment

Two experimental themes were selected, similarly to the case of GAS. In so doing, themes that were promising due to likely spin-offs were given priority in view of the limited payloads of small rockets. The development of experimental equipment was the responsibility of the Mechanical System Promotion Association, as was the case with GAS.

##### 1. Theme 1 (Formation of Oxide Superconductive Material)

Contact between superconductive substances will be improved and critical current density increased by heating and partially melting a high-temperature superconductive material, dispersing it evenly, and cooling it under the microgravitational environment. The study of the experimental theme and the development of the equipment are the responsibility of Hitachi, Ltd.

##### 2. Theme 2 (On-the-Spot Observation of Process of Crystal Coagulation)

The mechanism of the process of crystal coagulation will be confirmed by heating a liquid crystal compound under the microgravitational environment, imaging the fluctuation of the temperature and the concentration of the liquid by a Mach-Zehnder interferometer under conditions involving a temperature gradient, and recording it using a video-camera. The study of the experimental theme is the responsibility of the Electrotechnical Laboratory, Agency of Industrial Science and Technology, and the Mitsubishi General Laboratories, while the development of the equipment is being handled by Ishikawajima-Harima Heavy Industries.

The plan proposes that experimental equipment be developed from the second half of fiscal 1987 to the first half of fiscal 1989, and that the launching and recovery be conducted by MASER No 4, scheduled for March 1990.

### Conclusion

Technical experts expect much of the effects of space environment utilization but, on the other hand, experimental data are currently lacking. Various means of space-using experiments are being studied, and it is important to secure as many opportunities to experiment as possible and to accumulate basic data. It is hoped that our society's experimental plan will be of some help toward attaining this end.

Part 2. Research on Compound Semiconductor Crystal Growth Using Micro-gravitational Environments [Section contributed by Kazuo Ishida, representative and managing director, Space Environment Utilization Research Institute]

### Introduction

The Space Environment Utilization Research Institute [SEURI] was established in April 1986, with the support of the Ministry of Industrial Trade and Industry, for the purpose of laying the foundation for electronic materials processing technology under space environments and contributing to the future advance of its practical use. Its proposed capital, to be paid over a period of 7 years, totals 5.6 billion yen. The shares by investors include 70 percent for Japan Key Technology Center and 5 percent each for six private firms (Ishikawajima-Harima Heavy Industries, Toshiba, NEC, Hitachi, Fujitsu and Mitsubishi Electric).

A total of 27 TDY researchers (including those doubling as such) from the investor companies conduct SEURI research at their offices set up in the laboratories of their respective companies, and these studies are supervised by the SEURI Research Headquarters. The initial plan at the time of its establishment was to participate in the West German space experiment projects D2 and D3 using the space shuttle but, with the delay in the shuttle operation program, SEURI switched to a plan to first use the small West German rocket TEXUS and the conduct D2 experiments. INTOSPACE was designated by the West German Government as the window to participation in these experiments by private companies, and in March 1987 we contracted to participate in six D2 experiments (scheduled for December 1991), while in April 1988 we contracted for four of the TEXUS experiments to be conducted in the fall of 1988.

The D-experiments comprise a serial project, sponsored by the West German Government, in which about 70 materials, fluid physics and bio-science experiments will be conducted for 7 days using a space lab (crew: about seven) developed by ESA. D1 took place in October 1985. TEXUS is an unmanned rocket carrying four to six experimental systems and measuring 40 cm in diameter and 4.5 m in length. It is launched to a height of 240 to 270 km and is recovered by a parachute after passing through microgravitational environments for 6 minutes. It has been used in 18 launchings since 1977; it is launched from a snowy field (Esrange) in Sweden once in spring and in fall every year. (Operated by MBB/ERNO, a West German firm.)

### Research Themes

The origin of crystal growth is in the so-called fluid state, i.e., melt, solution and gas, and, on the ground, convection is found due to slight differences in specific gravity. The two major factors that govern crystal growth are mass transfer and heat transfer, and each of these is strongly affected by convection. So, first grasping accurately the transfer velocities of atoms and heat in the state without convection is basic to computer simulation, etc., and fundamental to advances in research on crystal growth.

In themes involving the crystal growth caused in space experiments, it is, indeed, interesting to see how the growth velocity differs between that occurring in space and that on the ground. The importance of space experiments involves eliminating the disturbance caused by convection as much as possible and thereby eliciting the process by which the fluid solidifies. From the implication of this point of view and equipment that can be used, individual research themes were selected as follows:

Office No 1 (located in Ishikawajima-Harima Technical Laboratories)

Growth of melted solution of PbSnTe (temperature gradient furnace)

Component elements in melted solution of PbSnTe

Measurement of diffusion coefficients (ignition furnace)

Office No 2 (located in Toshiba General Laboratories)

Measurement of diffusion coefficients of impurities and component elements in InSb and GaSb melted solutions (ignition furnace)

Office No 3 (located in NEC Basic Laboratory)

Measurement of heat conductivity in InSb melted solution (ignition furnace)

Office No 4 (located in Hitachi Central Laboratories)

Microstructure control of GaSb/Ge eutectic crystals (gas cooled soaking pit)

Office No 5 (located in Fujitsu Atsugi Laboratories)

Solution growth of GaAs (ignition furnace)

Office No 6 (located in Mitsubishi Electric's Optical Microwave Device Laboratory)

Gas-phase growth of InP (temperature gradient furnace and improved ignition furnace)

Joint Research Office (located in Ishikawajima-Harima Technical Laboratories)

Wettabilities of melted solutions (furnace: not yet decided)

Major Projects and Researchers

Our approach to individual research themes is changing considerably so as to conform to the shifting conditions of equipment that can be used for space experiments and the ground support setup. The process of matching the infrastructure of space experiments and the needs of researchers is probably most important to the development of space environment utilization and is

difficult. Therefore, the following is a description of our experiences (still uncompleted) concerning this process.

To a researcher, thinking of a theme after having been allotted existing equipment is like having his feet compressed to make them fit new shoes; it is difficult. Management by an organized group of users, as happened in our case, has the decided advantage of being able to increase the degree of freedom of researchers and, eventually, the certainty of success. For instance, the measurement of heat conductivity in melted solutions is technically difficult with the shuttle-mounted furnace, but we plan to accomplish this in our TEXUS experiment using a furnace we fabricated.

We have had various experiences in compromising on what furnaces to adopt for the D2 project. At first, the melted solution crystal growth experiment using a temperature profile that shifts with time was proposed from a number of offices. In D2, a three-band temperature gradient furnace (CNES, France, product) and a quenching temperature gradient furnace (Dornier System, West Germany, product) are likely to be used for experiments of this type but, although the former can test three samples at a time, the latter can handle only one sample. The outside diameter of the cartridge used is 24 mm for the former, but 9 mm for the latter. The latter does not permit free control of the temperature gradient and cooling velocity because it provides a temperature gradient by quenching the sample from its end. For these reasons and due to other catalog characteristic differences, preference was concentrated on the former. More than 20 people, including the D2 administrator of the West German aerospace research institute, the ground support manager, engineers from manufacturers of furnaces already mounted, engineers from the national space research center of France, INTOSPACE officials concerned and our group met three times to discuss the allocation of furnaces to be used. The procedure followed was this: We experimenters submitted our required specifications for equipment in advance. In response, the engineers from the manufacturers of furnaces already mounted explained the aptitudes of their furnaces in a conference. Then, the D2 administrator concluded with some overall coordination for the project. We had to change our initial plan since we learned during the adjustment that, regarding the use of the three-band temperature gradient furnace, we would be allotted only one or two experiments, and that it was complicated to shift the temperature profile by the heater input control of each band. In addition, we learned that it was necessary to buy a simulator furnace, and that we should familiarize ourselves with its characteristics.

These experiences indicate that, when forming a plan for an advanced experience, the researchers must engage in mutual direct discussion regarding equipment and samples, and it is extremely dangerous for them to rely on information exchange by catalogs or facsimiles. These technical matters were discussed frankly, but the process of coordinating the entire project was complicated. On one occasion, when we were suddenly asked to study the possibility of using a furnace that was technically unsuitable, we were able to get the furnace rejected by submitting our technical view, reached after arduous study,

that it was inexpedient. In the background were factors related to the United States, such as the amount of NASA-participation in experiments, total payload weight and the principle of safety screening, problems involving France which included the ground support setup for a French-mounted furnace, and the fact that preparations had to be made before the determination to who should pay the expenses of private participants and other matters, which were external factors affecting the D2 project administrator, were finalized. These problems involving international projects remain to be solved. Everyone is aware that major projects always require their participants to use limited resources fairly and in the spirit of mutual concession but, it is important to structure the operation so that participants can see what concessions are necessary for settlement. It is preferable that the decision-making for all matters vital to attaining the targets of a project, the assignment of roles and the personnel responsible for these roles be defined before the start of the project. However, if this is impossible due to external factors, it will be necessary to use as large a cooperation group as possible and form a setup permitting flexible operations.

#### Conclusion

Our first space experiment was conducted on 28 November 1988, using TEXUS. We sincerely hope that our trial-and-error experience report will be used effectively in preparing for the Free Flyer, space station and other future Japanese uses of space environments.

Clinical Testing To Start on Stem Cell Growth Stimulator

43073909a Tokyo JAPAN CHEMICAL WEEK in English 8 Jun 89 p 4

[Text] Otsuka Pharmaceutical Co. will begin clinical testing on the actions of interleukin-1  $\beta$  (IL-1 $\beta$ ) with regard to growth of stem cells. IL-1 $\beta$  has been produced by Otsuka as an antimalignant-tumor agent by means of DNA recombination. The substance is known to help stimulate growth of stem cells.

A stem cell is an unspecialized cell that gives rise to differentiated cells, e.g., red blood corpuscles, leukocytes, macrophages, blood platelets and lymphocytes (B-cells, T-cells). R&D has been under way on various interleukins acting to stimulate the differentiation of stem cells but these stimulators work properly only when the stem cells concerned are in a normal condition. Problems arise when the stem cells themselves are damaged by chemotherapy and radiotherapy or when their self renewal is insufficient.

The researchers involved now consider it important to use an agent stimulating the growth of the stem cells in combination with the said differentiation stimulators. A blood platelet-source substance has recently proved to stimulate growth of stem cells and it has also been confirmed that IL-1  $\beta$  has such an action.

Syntex Inc. of the U.S. succeeded in cloning interleukin-1 $\alpha$  and -1 $\beta$  in 1985 and Otsuka was successful in cloning IL-1 $\beta$  the following year. The Japanese firm has been carrying out R&D on both the blood platelet-source stimulator and IL-1 $\beta$ . The company is also considering conducting clinical testing on IL-1 $\beta$  in the U.S.

## BIOTECHNOLOGY

### Biotechnology Research Themes Selected by MITI for FY 90

43073909b Tokyo JAPAN CHEMICAL WEEK in English 22 Jun 89 p 1

[Article: "3 Biotechnology Research Themes Picked Out: MITI"]

[Text] MITI has selected development of chromosome-engineering technology, sugar-chain designing technology and biodegradable plastics as three themes for biotechnology research.

The three projects will be inaugurated next fiscal year with development terms and funds set at 10 years and ¥15 billion in each case.

They are aimed at controlling cellular functions by elucidating the chromosome, synthesizing artificial sugar chains by examining natural ones and producing macromolecular material having no harmful ecological effects, respectively.

MITI regards 1989 as being the first year for 2nd-generation biotechnology.

**Energy Agency's 'Moonlight Project' Developments**

43063539 Tokyo DENKI TO GASU in Japanese Feb 89 pp 13-20

[Article by Hidenobu Nakagawa, Moonlight Project Promotion Office]

**[Text] 1. Inauguration of Moonlight Project**

In the midst of a situation in which the solution of the energy problem was regarded as an urgent question from the viewpoint of the national economy after the first oil shock stemming from the fourth Middle East War in 1973, the Moonlight Project was inaugurated to contribute to the solution through the development of energy conservation technology. In order to promote the Moonlight Project, the Moonlight Project Promotion Office was set up in 1978 in the Agency of Industrial Science and Technology [AIST].

The Sunshine Project was inaugurated in 1974 to develop new energy (oil alternative energy). Based on the inauguration of the Moonlight Project, a structure was built up to promote conversion from oil for energy in Japan through technological development.

The energy situation at that time was that, with the first oil crisis in the autumn of 1973 as a turning point, the era of both abundant and inexpensive oil for energy, which had supported the world's postwar prosperity, ended and the world's energy situation was moving toward supply instability and high price. At the time of the first oil crisis, oil prices increased from \$3 to \$12 per barrel, and in 1978 it went up to \$34 due to the second oil crisis brought on by the Iranian Revolution. The influence exerted on the world economy by the second oil crisis was remarkable: it caused worldwide inflation, and many countries adopted tight policies to control inflation, which caused a grave business recession.

Such a situation prompted AIST to set up in 1978 a new system, "The Energy Conservation Technology Research and Development System (Moonlight Project)," calling for adoption as a national project the development of energy conservation technology ranging from basic research to the development stage as a means of comprehensively promoting energy conservation technology R&D and also actively utilizing existing policy, such as amplifying assistance to energy conservation technology research in the private sector.

Table 1. FY 1989 Moonlight Project Draft Budget

(Unit: Million yen)

Item	FY 1988 budget	FY 1989 draft budget	Outline of FY 1989 project
1. Large-Scale Energy Technology R&D	9,491 [422] [ESA 9,070 [BSA 0]	10,109 [351] [ESA 9,758 [BSA 0]	
(1) New type cell power storage system	2,027 [64] [ESA 1,963]	1,953 [38] [ESA 1,915]	To manufacture 1,000 kW new type cell power storage system pilot plant. Also, to conduct new type cell system test using simulated power transmission system. In addition, to conduct concept design to satisfy performance requirement of new type cell as a cell for electric motor vehicles.
(2) Fuel cell power-generation technology	3,550 [111] [ESA 3,439]	3,696 [78] [ESA 3,618]	To conduct comprehensive technology development of 200 kW class ceramic type power-generation system regarding phosphoric acid type fuel cell. Also, to conduct development of stack enlargement technology regarding a fused carbonate type fuel cell, and basic research regarding solid electrolyte type fuel cell.
(3) Super heat pump energy accumulation system	1,678 [141] [ESA 1,737]	1,791 [75] [ESA 1,715]	To conduct operational research on a bench plant consisting of 100 kW class high-performance pressure type heat pump and 10,000-ton-class chemical heat accumulation equipment. Also, to initiate the manufacture of 1,000 kW-class pilot system.
(4) Superconductivity electric power application technology	1,652 [67] [ESA 1,585]	1,952 [93] [ESA 1,870]	To conduct R&D of technology to materialize wire rod of superconductivity material of compound and oxide and of an element technology/system technology of superconductive generator. Also, to conduct research on a total system, such as a study on a test method of a superconductive generator machine.
(5) Ceramic gas turbine	384 [39] [ESA 345]	707 [67] [ESA 640]	To continue to conduct development of element technology of heat-resistant ceramic material manufacturing/processing technology. Also, to initiate trial manufacture of 300 kW-class basic type gas turbine.
2. Leading Basic Energy Conservation Technology	154	155	To conduct R&D of 11 themes including research on measuring heat-flow technology.
3. International Research Cooperation Projects	35	95	To continue to participate in IER improved type heat pump system implementation agreement and conduct binational cooperation between Japan and France. Also, to conduct research and cooperation on fuel cell utilization/area power generation system with Thailand (projected by International Trade Policy Bureau).
4. Survey on Energy Conservation Technology Establishment	7	6	To arrange and present R&D problems involving great energy conservation effect.
5. Subsidy for Energy Conservation Development in Private Sector	236 [38] [BSA 198]	360 [0] [BSA 360]	To provide subsidy to the R&D of energy conservation technology (heat pump system using Stirling engine) conducted by private sector (projected by Technology Promotion Section of AIST).
6. Energy Conservation Standardization	21	5	To conduct survey and research necessary for standardizing energy conservation, and also to build up the JIS standard related to energy conservation.
7. Others	14 [11] [BSA 3]	9 [9] [BSA 0]	Coordination expense of R&D projects and the administrative expense necessary for R&D.
<b>Total</b>	<b>9,957 [686] [BSA 9,073 [BSA 198]</b>	<b>10,739 [621] [BSA 9,758 [BSA 360]</b>	<b>■ Evaluation expense: Y589 (391) million (The total of electricity special account related to research institutes)</b>

ESA: Electricity special accounting

BSA: Oil special accounting

## II. Necessity of Energy Conservation Technology Development

Energy conservation means that while various social demands, such as enhancement of the standard of living and maintenance of employment are met, waste should be avoided in each stage of energy consumption, with energy being used as efficiently as possible.

At the same time, it means reducing the degree of oil dependence, environmental pollution, and energy costs.

Also, internationally, at the IEA (International Energy Agency) and the major advanced nation summit conference, etc., further energy conservation is being emphasized. The importance of the leading world powers conserving energy from the viewpoint of meeting worldwide demand was stressed.

In order to promote energy conservation policy, together with legal measures on rationalization of energy utilization and the development and spread of energy conservation enlightenment activities, the role to be played by energy conservation technology development, such as efficiency enhancement and waste heat utilization in the fields and conversion, transportation, storage, and consumption of energy, has become very great.

For example, in the case of electric power, together with consumers' efforts to save electricity, energy utilization efficiency must be enhanced when primary energy--oil and coal--is converted to electric power, but this is indeed a problem of technological development. Also, there still seems to be much room for technological development to convert to energy conserving equipment without loss of effectiveness.

On the other hand, energy conservation R&D technology has the following diversifications:

- the subject technology ranges from software to hardware;
- the R&D problem ranges from basic research to practicalization/improvement research;
- the R&D scales, such as the necessary time and the necessary funds vary;
- the users of results exist in many fields of national economy.

Therefore, it is necessary to promote R&D systematically and efficiently while combining appropriate R&D measures with structures.

The Moonlight Project to promote energy conservation technology R&D comprehensively and systematically while consolidating the forces of national research institutes, the industrial world, universities, etc., has been promoted based on the following six pillars at present:

- large-scale energy conservation technology R&D;
- leading/basic energy conservation technology R&D;
- international research cooperation on energy conservation technology;
- survey on the establishment of energy conservation technology;
- subsidy for energy conservation technology R&D in the private sector;
- promotion of energy conservation through standardization.

### III. Large-Scale Energy Conservation Technology R&D

Because the development of large-scale technology has a remarkable energy effect and also is urgently needed, it nevertheless involves great risks requiring massive funding and a long R&D period. If it cannot be promoted

by the private sector alone, it will be conducted through the technological development organically combining the R&D at national research institutes with that in the private sector. The R&D of the following projects are being promoted:

**(1) New-Type Cell Electric Power Storage (FY 1980-81)**

Because it is difficult to store electricity, unlike oil and gas, it is necessary to adjust supply, namely power generation, in accordance with power demand changing every hour. Also, the range of fluctuation in power demand, the annual generated power quantity and the maximum power demand needed in the daytime in summer have become greater year after year because of the enhancement of the standard of living. For this reason, it is necessary to maintain power plants on standby to immediately respond to electric power fluctuation and to secure a power-generation system in response to the maximum power demand, but this is rather wasteful from the viewpoint of efficient operation of a power-generation system and effectiveness in system investment. Also, power plants have increased in size year after year, but it is necessary for these large-size power plants to generate power stably while maintaining specific output in order to retain high efficiency. Therefore, a request has intensified toward electric power storage technology to store electric power at night and supply it in the daytime when demand increases as a way to meet demand fluctuations (such an averaging of power load is called load leveling).

For this purpose, the R&D of a "new-type cell power storage system" was initiated in 1980 as one of the large-scale energy conservation technology R&D projects of the Moonlight Project.

Completion of this technology will permit promoting effectiveness in energy conservation through efficient operation of the power-generation system and enhancement of power generation efficiency; it will also permit promoting alternative power sources using fuel other than oil. Moreover, as this power storage system promotes preserving the environment and can be installed in cities, it is possible to decrease transmission power losses and to reduce the capacity of power transmission lines. Also, great hopes are being placed on the system as one enabling the stable supply of the power generated from natural energies, such as solar energy, wind power, tidal power, etc.

The R&D of a new-type cell power storage system, a new-type cell, and of a system technology, and also the power storage system verification test integrating the two will be conducted; moreover, practicalization is scheduled to be realized by FY 1991.

**(2) Fuel Cell Power-Generation Technology (FY 1981-95)**

Electric power, accounting for about 30 percent of energy demand in Japan, has been widely used in such fields as industry, the home, and transportation because it is convenient, clean, and safe. The demand for power is expected to increase more and more in the future.

However, in thermal power generation to supply the electric power, power-generation efficiency is only about 40 percent, the remaining 60 percent being wasted. For energy conservation, the development of highly efficient power-generation technology has become a social problem.

Because the fuel cell power-generation system is very efficient, regardless of its scale and natural gas, methanol, coal gas, etc., can be used as fuel, it is possible to promote high efficiency in the power-generation branch and diversification of fuels replacing oil. Also, because the system does not cause much air and noise pollution and is superior in preserving the environment and also can be installed at the power demand area, it is possible to reduce power transmission losses.

This project calls for developing four kinds of fuel cells: centering on the phosphoric acid-type fuel cell, on which great hopes for early practicalization are placed, the fused carbonate-type fuel cell, the solid electrolyte-type fuel cell, and the alkali-type fuel cell.

### (3) Super Heat Pump Energy Accumulation System (FY 1984-92)

At present, about 50 percent of all the energy used in Japan has been emitted to the environment in the forms of waste gas, cooling water, etc. without being effectively used.

It is difficult by the present technology to recover and utilize waste heat whose difference in temperature from the environment is especially small or whose fluctuation in volume is great. Therefore, the waste heat is not used.

It will be important to effectively recover waste heat exhausted in such great volume and to use it effectively for other purposes in order to reduce energy use in Japan.

Also, from the aspect of energy supply, it is effective to efficiently conduct so-called load leveling so that surplus power at night can be effectively used and also promote reduction of peak power demand in the daytime.

This project calls for developing a system combining a super high performance pressure type heat pump with chemical heat accumulation technology and conducting heat accumulation by using base supply electric power at night with oil alternative energy as its power source. This project also calls for reducing peak electric power in the daytime and at the same time contributing to averaging the load rate by recovering very efficiently and utilizing low grade waste heat not yet used in air conditioning/hot water supply in the daytime and at an oil refinery, etc.

### (4) Superconductivity Electric Power Application Technology (FY 1988-95)

Superconductivity is a phenomenon in which continued cooling of a certain kind of material, suddenly makes electric resistance drop to zero. Also, if a magnetic field is added from outside, it has the property of erasing

the internal magnetic field (Meissner effect). Material having this property of superconductivity will not cause loss of electric resistance if electricity flows through it, but will be able to carry a much higher density of electric current than ordinary conductive materials such as copper.

At present, as to superconductive materials, metal family materials (niobium titanium alloy, niobium 3 tin compound, etc.) requiring cooling by liquid helium (temperature: about -269°C) have been practicalized as wire rod, etc., but in the future, expectations are being placed on the development of producing wire rod by high-temperature superconductive material of the oxide family available at more than liquid nitrogen temperature (about -196°C).

On the other hand, in order to meet an increase in power demand, additional installation/expansion of power-generation facilities and power transmission-transformer facilities are being promoted, but problems in the difficulty of securing land for power transmission lines and in the stability of the electric power system are appearing on the surface as power sources become large in capacity and are set up in remote places. In order to deal with such problems efficiently, it is necessary to introduce superconductivity electric power application equipment, such as superconductive generators, and to promote application of high efficiency and high stability in the electric power system.

For this purpose, this project calls for conducting R&D for a superconductive wire rod, for a total system, and for a freezing system centering on the development of superconductive generators (70,000 kW-class).

#### (5) Ceramic Gas Turbine (FY 1988-96)

Heat efficiency of small generators being used for cogeneration and small-scale power generation in Japan is no more than about 35 percent, but if a ceramic gas turbine is developed, 40-45 percent heat efficiency can be achieved. Also, as ceramic is a superior material against heat and corrosion, it will be possible to increase turbine inlet temperature to 1,300-1,400°C and to acquire high-temperature exhaust air. Therefore, the ceramic gas turbine is considered to be promising as a generator for cogeneration.

This project is to develop cogeneration and a 300 kW-class ceramic gas turbine for power generation and to contribute to the promotion of energy and space conservation and diversification of power sources in the field.

For this purpose, this project will conduct R&D for anti-heat ceramic material and element technology. Design/test operation of a 300 kW-class ceramic gas turbine will be conducted and also the research on social adaptability, such as environment influence measuring, will be conducted.

On the other hand, the four projects completed are as follows:

### (1) Waste Heat Utilization Technology System (FY 1976-81: conducted)

About 60 percent of all energy consumption in Japan is accounted for by the mining and manufacturing branch. Moreover, about 50 percent of the energy consumed is exhausted to air, river, sea, etc., as waste heat. Therefore, in order to promote energy conservation, it is very important to recover heat exhausted from industry and to utilize it effectively. From this viewpoint, the research of a system to effectively recover waste heat from plants, etc., and to use it effectively was set as a goal. For this purpose, the R&D of element technologies such as heat recovery/heat exchange and heat transport/heat storage and of a system to utilize these comprehensively was conducted.

Since FY 1982, efforts have been made to promote widespread diffusion of the results acquired by the research done so far. Also, as a result of R&D, the desired results, such as development of the absorption-type heat pump system, were acquired. Patent applications on 99 items were made, and, as of March 1987, registration of 72 items, utility models of 17 items, and accumulation of a number of know-hows were acquired. At present, these are already being put to practical use at scores of plants in Japan and exported to foreign countries, too.

### (2) Magnetohydrodynamic (MHD) Power Generation (FY 1966-83: conducted)

At present, thermal efficiency of the existing steam power generation is said to be 40 percent at the most, but the MHD power-generation method is to enhance this to about 50 percent. A considerable energy conservation effect is expected to be obtained.

During the first period from FY 1966-75, the R&D of component equipment, such as a superconductive magnet, etc., produced the desired results. After that, during the second period in the Moonlight Project, the research was conducted to develop MHD power generators to meet technological conditions as regards durability and power-generation output. The Mark VII experimental generator with a capacity of 100 kW output was manufactured and 430 hours of power generation could be done. Results on verification of durability of power generation channels through kerosene burning were acquired, and design data necessary for building the next pilot plant (100 Mwt) were accumulated.

As to coal burning, R&D of the leading basic energy conservation technology has been conducted since FY 1984.

### (3) High Efficiency Gas Turbine (FY 1978-87: conducted)

A combined cycle power-generation system combining a gas turbine with a steam turbine driven by high-temperature steam obtained from the air exhausted from the turbine will remarkably enhance overall thermal efficiency. This R&D developed a reheating type gas turbine suitable for a combined power-generation system and to attain overall thermal efficiency of 55 percent.

In a mid-stage of the R&D, a pilot plant (overall thermal efficiency: 50 percent; output: 100,000 kW; turbine inlet temperature: 1,300°C) incorporating the results of the development of material technology and element technology was manufactured.

An output efficiency of 52 percent was attained through operational research. In addition, a total operation time of 548 hours and total power generation of 18.21 million kWh were recorded. The test was completed in July 1987. In the R&D of the prototype plant in the final stage, it has become possible to attain a turbine inlet temperature of 1,400°C through a high-temperature high-speed turbine test and 55 percent overall thermal efficiency was obtained.

At present, gas turbine combined cycle power-generation is steadily spreading as a high-efficiency power-generation system. Element technologies developed by this R&D, such as cooling methods of heat-resisting alloy, heat-resisting ceramics material, burner, and turbine, have begun to be used.

#### (4) General-Purpose Stirling Engine

The Stirling engine is a kind of external combustion engine producing rotary power by heating/cooling high-pressure gas confined in a cylinder from outside of the cylinder. This engine has characteristics suitable for energy conservation and alternative energy use. Compared with the internal combustion engine, thermal efficiency is high, the engine is quiet, and various kinds of fuel, including natural gas, coal, and wood can be used.

In this project, the R&D on Stirling engines of the 3 kW-class and 30 kW-class and the heat pump system for air conditioning suitable for Stirling engine and small-scale generator system has been conducted. As a result, the desired performance goal was acquired and the basis toward the practicalization of the Stirling engine could be set up.

At present, in order to commercialize medium- and small-size heat pumps for air conditioning, the research on the enhancement of durability/reliability and cost reduction is being promoted mainly by private companies.

### IV. Leading/Basic Energy Conservation Technology R&D

The R&D on leading technologies and basic technologies, such as technologies to be seeds of energy conservation technology in the future and system technologies to be applied to new fields by effectively combining various element technologies, difficult for the private sector to handle, is being conducted by various research institutes under AIST.

In FY 1988, the R&D of nine themes such as combined type reaction equipment equipped with high-functional separation membrane and MHD power-generation technology will be conducted.

#### **(1) Research on Measuring Technology of Heat flow**

In order to control thermal energy to be lost from such heat equipment as a high temperature furnace to the minimum and to operate it efficiently, it is necessary to accurately understand the thermal properties of material constituting heat equipment. For this purpose, various methods to enable to accurately measure the thermal properties (thermal conductivity, ratio, radiation rate) of adiabatic material at high temperature will be set up.

#### **(2) Research on Energy Conservation-Oriented Manufacturing Methods of High Fusing Point Inorganic Compound**

In recent years, in the manufacture of ceramic material, for which demand is increasing, instead of the conventional method consisting of two processes of synthesis and sintering, the R&D of high efficiency manufacturing technology to conduct synthesis and molding simultaneously in one process through self-exothermic reaction will be conducted.

#### **(3) Research on High Efficiency Power Conversion Technology Through Re-Thermal Cycle**

To promote high efficiency and low pollution by internal combustion engines, the R&D of a hybrid engine consisting of an adiabatic diesel engine and a gas turbine engine will be conducted. The technology to effectively utilize high temperature heat exhausted from an adiabatic diesel engine and to recover power efficiently will be set up. The technology to enable low pollution through burning control by a re-thermal burner will be setup, thus promoting energy conservation.

#### **(4) Research on Upgrading Burning Technology**

In order to promote energy conservation by reducing the loss of exhausted gas heat in industrial burning equipment, the technology to swiftly examine the burning condition of the burner flame and the control technology to optimize burning under the various operational conditions will be developed. At the same time, research on an energy conservation-type wet-coal-burning technology to use coarse powder coal is being conducted.

#### **(5) Research on Complex-Type Reactor Equipped With High Function Separation Membrane**

In order to promote energy conservation in the chemical industry, R&D on a complex-type reactor equipped with a high function separation membrane will be stepped up.

Through this technology, as the easing of equilibrium chemical reaction condition under high temperature and the enhancement of reaction rate can be promoted, high efficiency in manufacture of chemical products will become possible.

#### (6) Research on Coal-Burning MHD Power Generation

MHD power generation is a direct power generation method to take the power generated by means of piercing the burning gas plasma acquired through high temperature burning of fossil fuel (oil, natural gas, coal) at high speed into a power-generation channel place in the powerful magnetic field. By setting up a MHD-steam complex power-generation system combining MHD with a steam turbine power-generation system utilizing the exhausted heat, efficiency will be enhanced to more than 50 percent.

In this research, from the viewpoint of coal utilization, the development of a coal burner peculiar to coal-burning MHD power generation and of a power-generation channel, etc., are being conducted.

#### (7) Research on Natrium Heat Electricity Conversion Technology

Research on technology to convert thermal energy directly to electricity by making use of ceramics ( $\beta$ " alumina) in which  $\text{Na}^+$  ion flows is being conducted.

This is to generate electricity by the flow of the  $\text{Na}^+$  ion from a high-temperature surface to a low-temperature surface of  $\beta$ " alumina based on a natrium vapor pressure difference caused by temperature difference.

As this power-generation method has great output density and high power generation efficiency, expectations are placed on it as a small-size, high efficiency power source.

#### (8) Research on Energy-Oriented Manufacturing Technology of Fine Ceramics Materials

Chaff carbide consisting of noncrystalloidal  $\text{SiO}_2$  and carbon is very responsive and is a promising substance to make it possible to manufacture fine ceramics materials such as  $\text{Si}_3\text{N}_4$  powder, etc., by using a small amount of energy. However, as it is a minute particle, it has a property of bad fluidity. There was a problem of obtaining a high productivity in nitrogenization reaction at the fluid bed. In order to solve this problem, the R&D of energy conservation-oriented ceramics manufacturing technology by using a vibration fluid bed of high temperature is being conducted.

#### (9) Research on High Lithium Conductivity Solid Electrolyte

Lithium is a substance which has strong activity and is suitable for light and high-performance batteries. This research is for a lithium ion solid electrolyte with high conductivity necessary for the development of a lithium secondary cell of high power density, high energy density, and long life, and for an ion electron mixed conductor to be used for the electrode.

### V. International Research Cooperation Basic Research

In order to promote the R&D effectively, it is always important for us to understand appropriately the actual situation of R&D at home and abroad and

the trend of related technologies and also for many countries to cooperate and conduct such R&D as necessary. In particular, the energy problem in recent years is a problem common to many countries in the world. In order to meet increased long-term demand for energy in the world, it is indispensable to have international technical cooperation.

In IEA also, nine action agreements have been concluded in the energy conservation field. In the agreements, many projects are being promoted. Japan is participating in the "improved type heat pump system agreement," and is promoting research and cooperation.

Also, Japan has concluded binational scientific technical cooperation agreements with the United States, France, etc., and based on the agreements, cooperation and information exchanges have been conducted, mainly in the energy conservation field. Among these, in the cooperation between Japan and France, a place for exchanging opinions by experts of the two countries is to be provided.

#### **VI. Survey on Establishment of Energy Conservation Technology**

In order to promote energy conservation effectively, needless to say, it is important to conduct technological development of equipment/plant, etc., but at the same time, it is necessary to dig up energy conservation technology development problems from a medium- and long-term viewpoint and also to understand the relationship with technologies related to the problem.

For this purpose, the survey to set up a comprehensive method to gain effectiveness of energy conservation technology was initiated in FY 1982. This involves digging up energy conservation technology development problems by using a technology correlation table of "problem vs. technology" and "technology vs. element technology," collecting energy data in the broad fields, such as electricity, chemistry, machinery, biology, and nature, and also understanding the effect of technology development in each stage--resources, energy conversion, transportation, etc.--through the total energy flow model.

#### **VII. Subsidy for Energy Conservation Technology Development in Private Sector**

To especially important energy conservation technology research projects conducted by private enterprise, etc., the state will provide subsidies to cover part of the research expenses based on the energy conservation technology R&D expense subsidy system and oil alternative energy technology practicalization development expense subsidy system. In doing so, smooth promotion of the R&D concerned is intended.

Table 2. Long-Term Development Plan on Large-Scale Energy Conservation Technology R&D

## VIII. Promotion of Energy Conservation Through Standardization

Energy conservation through standardization is to promote energy conservation through enactment, revision, etc., of the Japanese Industrial Standard (JIS) from the viewpoint of energy conservation and also through utilization of the JIS mark (◎) display system indicating that quality, performance, etc., of the mining and manufacturing industry products concerned are in accord with these standards. For this purpose, standards to help promote energy conservation will be actively enacted and revised. At the same time, the survey and research to acquire basic data necessary for building up such standards are being conducted.

**NEC's SOR Lithography R&D Described**

43064030 Tokyo OPTRONICS in Japanese Mar 89 pp 118-123

[Article by Koichi Okada, NEC Corp.: "Atmospheric Exposure System SOR Lithography Technology"]

[Text] 1. Foreword

In recent years, the design rule for semiconductor devices has come into the submicron domain. In the mass production line, lithography carries much weight. In order to overcome the limits of resolution due to diffraction of light, the light source to be used has become shorter in wavelength. In the 0.6~0.7  $\mu\text{m}$  line width region, the optical exposure using the g beam (wavelength 436 nm) and the i beam (wavelength 365 nm) of mercury is considered to form the mainstream. In the smaller line width region down to 0.4~0.5  $\mu\text{m}$ , the excimer laser (for instance, KrF of 248 nm wavelength) exposure is regarded as promising. In the half-submicron line width region of 0.5  $\mu\text{m}$  or below, SOR (synchrotron orbit radiation) lithography is most promising.

SOR lithography is considered to be the most promising batch exposure system transfer technology that replaces the conventional optical exposure in the line width region of 0.5  $\mu\text{m}$  or below.<sup>1</sup> As lithography, soft X-ray beams of 0.5~1.5 nm wavelength are used. the high intensity and the high parallelism of SOR are arousing hopes for high throughput and high resolution as lithography technology. The SOR lithography has the following features: 1) high resolution approaching even 0.1  $\mu\text{m}$  (in the experiments, up to the resolution of several hundred  $\text{\AA}$  has been confirmed); 2) a large depth of focus of  $>\pm 10 \mu\text{m}$  ( $\pm 1 \mu\text{m}$  or below in excimer laser); 3) a wide exposure range of several centimeters square; 4) the use of single-layer resist of simple process; and 5) a high throughput. Consequently, it is positioned as a promising lithography candidate for producing large-capacity memories of the 64-Mb or 256-Mb class, which are expected to be put into mass production after the mid-1990s.

SOR lithography is now drawing world attention. At the 34th National Symposium of the American Vacuum Society, which was held from 2-6 November 1987 in Anaheim in the United States, a session entitled X-Ray Lithography With Synchrotron Radiation was arranged, and the newest pieces of information on SOR lithography in the United States, West Germany, and Japan

were introduced. It was reported that in West Germany, there are good prospects in a series of R&D projects, including the subminiature SOR light source (called COSY), the X-ray photomask, and the X-ray photoresist. In the United States, studies on the lithography system, including the vertical stepper development, are being carried out, using the SOR light source (Brookhaven) of the NSLS (National Synchrotron Light Source). In addition, SOR irradiation effects (such as stress and dimension changes) on the X-ray photomask are being actively researched, using the SOR light source (Aladdin) of the SRC (Synchrotron Radiation Center, Wisconsin University) and the SOR light source of the SSRL (Stanford Synchrotron Radiation Laboratory).

In Japan, the development of subminiature SOR light sources is now being conducted actively.<sup>2</sup> In addition to one such light source (named Niji [Rainbow]) through joint development by the Electrotechnical Laboratory and Sumitomo Electric Industries, Ltd., a subminiature SOR light source (named AURORA) using a new implantation method that was developed by Sumitomo Heavy Industries, Ltd., and a concept of SOR facilities for multiple purposes through joint development by the National Laboratory for High Energy Physics [KEK] and Furukawa Electric Co., Ltd., several other projects are being pushed simultaneously. As the SOR light sources being used for SOR lithography research, there are now TELL-TERAS of the Electrotechnical Laboratory, and PF (Photon Factory, synchrotron radiation experimental facility) of the KEK. In the PF in particular, the lithography beam lines of several domestic makers have been constructed, and active R&D is being carried out.

Recently, IBM has been conducting studies on test manufacturing devices by using SOR lithography. With regard to the nMOS of 0.5  $\mu\text{m}$  rule, it has applied SOR lithography to all of the eight levels and confirmed the performance of the element device.<sup>3</sup> The X-ray photomask consists of the Si membrane and the Au absorber pattern by the electrolytic plating method. In Japan, meanwhile, an SOR facility of NTT was completed last year, and tried immediately for test manufacturing devices. By forming 0.35  $\mu\text{m}$  patterns by SOR lithography in the Si MOSIC wiring layer, the operations of a test manufactured 51-stage ring oscillator were confirmed.<sup>4</sup>

During the past few years, NEC has been developing an SOR lithography system using the storage ring (electronic energy 2.5 GeV, maximum stored current 500 mA, orbit radius 8.66 m) of the PF of the National Laboratory for High Energy Phys. Especially, it is pushing atmospheric exposure system SOR lithograph hnology. Here in this paper, an outline of its activities, centering on equipment development, is to be introduced.

## 2. Themes, Development Policy

As mentioned above, there is completion for SOR lithography R&D among Japan, the United States, and Europe, and the remaining problems have also become clear gradually. In the aspect of equipment, they involve the development of an SOR lithography system, including a subminiature SOR light source, a beam line and a vertical SOR stepper. From the aspect of process, they involve the development of an X-ray photomask of the 0.25  $\mu\text{m}$  level that can

be put to practical use, and the development of a practical X-ray photo-resist with high sensitivity and high dry-etching resistance. Further, it is necessary to examine applications to test manufacture devices and the capability to resist damage.

Consequently, in order to put high-speed high-accuracy transfer technology at the  $\leq 0.25 \mu\text{m}$  level to practical use, it is necessary at any cost to set a definite target and to map out a detailed and long-range plan.

In order to put SOR lithography to practical use in the near future, it is considered that the construction of an SOR lithography system that is fit for practical use is necessary in the first place. It is especially important to attain the development of a subminiature SOR light source. There are two points for constructing a high-speed batch-transfer system SOR lithography system. The first point is how to expand the exposure range. SOR has a uniform intensity horizontally, but its uniform intensity range in the vertical direction is very narrow. In order to expand the vertical uniform exposure range, three techniques are being examined. They are to change the orbit of electrons around the SOR light source,<sup>5</sup> to use the oscillation mirror,<sup>6</sup> and to fix the photomask and the wafer and move them simultaneously.<sup>7</sup> Which technique is chosen has a decisive effect on the structure of the beam line and the exposure stepper. As the second point, there is the selection of the exposure atmosphere for transferring the photomask patterns to the wafer. Since SOR is a horizontally irradiated light, a vertical stepper comprising vertically arranged photomask and wafer must be used as its exposure stepper. The problem in this case is whether to set the vertical stepper in the atmosphere, in the He gas at the atmospheric pressure, in the decompressed air, or in the decompressed He gas. This must be examined from various points of view, such as the reliability of the mechanical system of the stepper, the attenuation of SOR in the exposure atmosphere, the temperature rise of the photomask, the effect on the alignment accuracy, the practicality, and the durability.

As a problem that has a considerable effect on the above-mentioned two points, there is the SOR leading-out window. As the SOR leading-out window, a several hundred  $\mu\text{m}$  thick He window is usually used for hard X-rays (wavelength  $\mu\text{m}$  thick Be window is usually used for hard X-rays (wavelength several Å or below) from the standpoint of its permeability to SOR, its strength as a window material, etc. For exposure use, it must be permeable to soft X-rays with a wavelength range of 0.5~1 nm, and therefore, a thin material with a thickness of several tens of  $\mu\text{m}$  or below is required. Should Be become thinner, it becomes permeable to X-rays with a wavelength longer to that degree, but its strength decreases gradually. Also, it must have a large area corresponding to the exposure range. Since the Be window is intended for leading out SOR from the ultrahigh vacuum beam line into the exposure atmosphere at a high pressure (atmospheric pressure), it must have a pressure resistance of 1 atmosphere or above.

The development policy, which was determined upon constructing the SOR lithography system, is described below. As a method to expand the exposure range, an oscillation mirror was used. The exposure atmosphere is the air

(1 atmosphere). Therefore, technology to lead out SOR into the air became necessary, and an original mechanism was developed.

Upon determining the atmospheric exposure system, the greatest importance was placed on the point from the aspect of alignment mechanism and system. In lithography, especially in transfer technology, what becomes the eventual key point is considered to be alignment accuracy. Consequently, fundamental as they are, early measures have been taken for research on the alignment method. Also, it was considered to be one point in the development to design a system that could more effectively display mass production capability as a great merit of SOR lithography. Thus far, most SOR lithography has not yet emerged from the stage of the vacuum exposure system or the decompressed He exposure system. In the case of these systems, it was considered that the development of practical equipment might be difficult in view of the following problems: the lubricant necessary for stable drive of the wafer-mask stage cannot be used; the equipment structure becomes complicated because a vacuum system mechanism is added to the precise mask-wafer mechanism; and the equipment is difficult to handle for operation and maintenance. Thus, taking early note of the atmospheric exposure system, R&D on it has been pushed steadily. The atmospheric exposure system is considered to have the following advantages: 1) lubricant can be used; 2) the vacuum system is not necessary, and the mechanism is simple; 3) the throughput can be improved because evacuation time is not necessary; and 4) operation and maintenance are easy.

Next, an oscillation mirror was used as the method to expand the exposure range. The method of causing changes in the electron orbit is considered to be unsuitable for industrial use because the SOR light source system becomes complicated in mechanism and structure. Meanwhile, the method of fixing the photomask and the wafer and moving them simultaneously imposes too many burdens on the alignment mechanism. Therefore, if the eventual alignment accuracy is considered, the realization of a high-precision alignment accuracy ( $0.05 \mu\text{m}$  ( $3\sigma$ ) or below) is considered to be fairly difficult. Thus it was concluded that the oscillation mirror is the most suitable method to expand exposure range. Also, since using the oscillation mirror, as compared with the method of fixing the photomask and the wafer and moving them simultaneously, imposes far smaller burdens on the alignment mechanism, a higher-precision alignment accuracy can be expected of it basically.

As a major technical theme for realizing the atmospheric exposure system, there is the development of an SOR leading-out window to lead out SOR (especially soft X-rays with a wavelength of 5~15 Å) from the vacuum into the air. In other words, in order that the window be fully permeable to soft X-rays, its material must be as thin as possible, and in order to make large the exposure range per shot, the window must be wide in area. Since this window must have the strength to resist a pressure of 1 atmosphere, it is a major technical theme to develop a thin and large-aspect-ratio SOR leading-out window.

### 3. Equipment Development

Figure 1 shows the structural diagram of the currently developed atmospheric exposure system SOR lithography system. The SOR light source is the storage ring of the synchrotron radiation experimental facility (PF) of the KEK. The beam line is an ultrahigh vacuum beam line having a fail-safe system to prevent a vacuum break. There is an SOR leading-out mechanism to lead out SOR from the ultrahigh vacuum into the air, and at the end of the beam line, a vertical SOR stepper comprised of the photomask and wafer set in the air is installed.

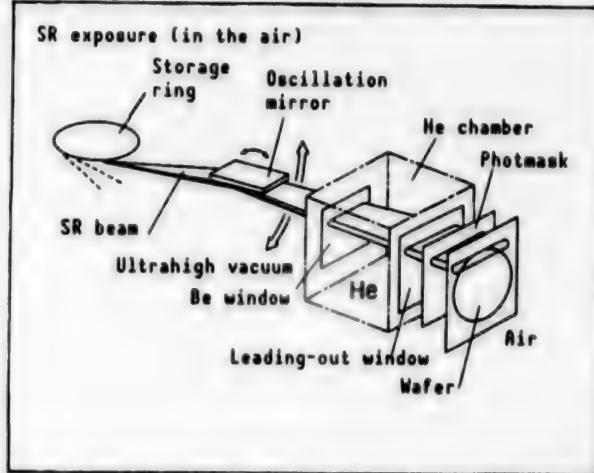


Figure 1. Structural Diagram of Atmospheric Exposure System SOR Lithography System

The developed beam line is a highly reliable SOR lithography beam line,<sup>6</sup> which is called BL-9A in the KEK-PF. The BL-9A is one of the three branch beam lines from the key channel BL-9. The BL-9A has a total length of about 13 m, and is structured up to the Be window under  $10^{-9}$  Torr ultrahigh vacuum specifications. In case of the Be window break, the fail-safe system was adopted. In other words, in the upper stream of the oscillation mirror, a high-speed cutoff valve with a closing time of <15 ms and a shock delaying tube of a delay time of 40 ms were installed. Figure 2 [not reproduced] shows an exterior view of the high-speed cutoff valve. The key channel BL-9 also has a combination of high-speed cutoff valve and shock-delaying tube. Thus, it is meant that there is eventually a double vacuum break preventive mechanism. The high-speed cutoff valve is a valve which, receiving signals of a pressure rise detected as a result of the Be window break, for instance, in the lower stream side of the beam line, effects closing at a high speed and prevents the air from entering into the further upstream side (the uppermost stream is the storage ring). The shock-delaying tube is effective for delaying the time of propagation of the air (pressure wave) that has come in due to the Be window break, etc.

As a key device of the exposure system, an oscillation mirror is installed in the beam line to expand the exposure range. The principal specifications are that: 1) the mirror material is SiC formed by the CVD method; 2) that the mirror size is 400 x 170 x 40 mm; 3) the mirror shape is plane or cylindrical; 4) the mirror drive is a combination of DC servomotor and cam mechanism; 5) the oscillation range is  $14.8 \pm 2-3$  mrad; and 6) the degree

of vacuum of the mirror chamber is  $10^{-9}$  Torr. Detailed study was carried out on the uniformity of exposure intensity using this oscillation mirror.<sup>8</sup> Measures were taken to control the scan speed for SOR as the function of the glancing angle of SOR for the mirror. By using a cylindrical mirror and applying optimum speed control to the cam drive, a uniformity of exposure intensity of  $>\pm 3$  percent was obtained in an exposure range  $25 \text{ mm}^2$ . Figure 3 [not reproduced] shows an exterior view (photograph of the inside scene) of the oscillation mirror.

Figure 4 [not reproduced] shows an exterior view of NEC's SOR lithography beam line BL-9A in the KEK-PF.

An SOR leading-out mechanism, which is comprised of a Be window, an He chamber, and a leading-out window as shown in Figure 1, was developed. It has a total length of about 52 cm, and a cylindrical shape of about  $15 \text{ cm}^2$  in average cross section. With regard to the Be window, immediate specifications were determined by carrying out fundamental studies on pressure resistance, etc.<sup>9</sup> By asking PF researchers to evaluate the preliminary studies on the Be window that this company carried out, tentatively the use of a Be window of a thickness of  $50 \mu\text{m}$  and a size of  $35 \text{ mm}\phi$  was permitted. The He chamber is filled with He gas, and put under pressure control of 1 atmosphere  $\pm 10 \text{ mm aqua}$ . The oxygen concentration in the He chamber is kept at  $<100 \text{ ppm}$ . The leading-out window is a  $1 \mu\text{m}$  thick silicon nitride film ( $\text{SiN}_x$ )  $26 \text{ mm}^2$ . Since outside the leading-out window is the air at 1 atmosphere, no pressure is applied to the leading-out window.

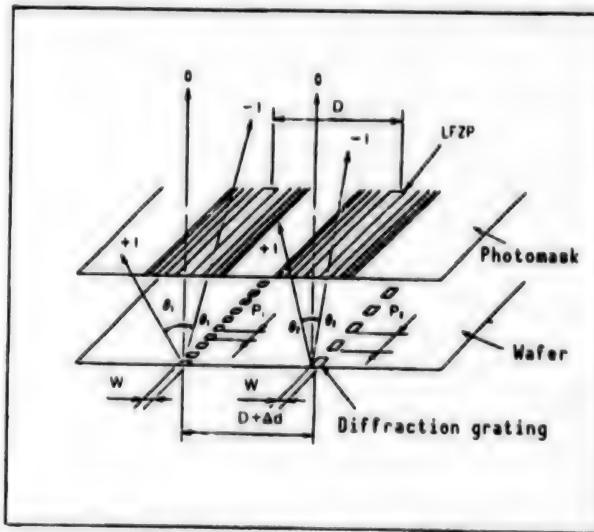


Figure 5. Diagram of Principle of Differential LFZP Method

A vertical SOR stepper using a new alignment method was developed.<sup>10</sup> For high-precision misalignment detection, it uses the "Differential Linear Fresnel Zone Plate Method" [LFZP] using two specific optical lenses, and a wafer and photomask driving mechanism that combines DC motor and piezo element, thereby making a high-precision alignment possible. Figure 5 shows the principle of misalignment detection by the differential LFZP method. On the photomask, two pieces of LFZP are formed by X-ray absorbing material, and their focal distance is equalized with the gap between the photomask and

the wafer. Meanwhile, on the wafer, two diffraction gratings of different pitch are formed, and their center-to-center distance is made larger by  $\Delta d$  than the distance  $D$  between the two pieces of LFZP.

Parallel laser beams applied from above are re-linearly collected by the LFZPs, and hit the diffraction gratings on the wafer. The laser beams reflected and diffracted here pass through the LFZPs again and return to the parallel beams, and are detected separately from the incident beams. The detection intensity becomes maximum when the LFZP center and the diffraction grating center correspond with each other. Thus, if the distance between the diffraction gratings differs by  $\Delta d$  from the distance between the LFZPs as shown in Figure 5, the peaks of the two detectors present a spatial phase difference of  $\Delta d$ . Consequently, by taking the difference between the two outputs, S-shaped misalignment signals can be obtained. Under the present circumstances, a positioning accuracy of  $0.03 \mu\text{m}$  and an alignment accuracy of  $0.05 \mu\text{m}$  ( $\delta$ ) have been acquired. Figure 6 shows a schematic diagram of the SOR stepper.

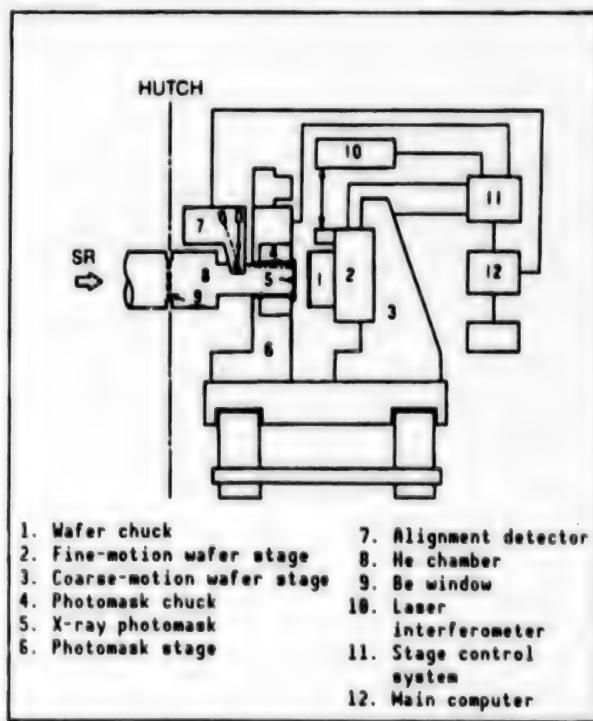


Figure 6. Schematic Diagram of SOR Vertical Stepper

With regard to the currently developed atmospheric exposure system SOR lithography system, detailed studies are now being carried out on such matters as alignment accuracy. Figure 7 [not reproduced] shows a transfer pattern of a  $0.2 \mu\text{m}$  width obtained by using this system.

#### 4. Future Course

The author should like to describe the future course of overall SOR lithography, although it may somewhat overstep the introduction to the

exposure technology entitled "Atmospheric Exposure System SOR Lithography Technology."

#### 4.1 SOR Lithography System

At present, practical SOR lithography systems are being developed mainly by West Germany, Japan, and the United States. The most important point is to develop a subminiature SOR light source. In connection with the development of a subminiature SOR light source exclusively for lithography use, such firms as IMT of West Germany, Sumitomo Electric Industries, Sumitomo Heavy Industries, and NTT of Japan, and IBM of the United States are resorting to their respective development strategies. As a judgment criterion to powerfully push the SOR lithography, it may possibly be enough to consider the prospect of attaining the development of a subminiature SOR light source. In other words, it is an idea that when a current storage of several hundred mA can be attained or a prospect of this being possible to acquire in a subminiature SOR light source of several meters in diameter, it would become a very important turning point toward practical use of SOR lithography. Meanwhile, there are also many problems to be solved in the beam line and vertical SOR stepper exclusively for lithography use.

#### 4.2 Overall Alignment Accuracy

In order to apply SOR lithography to device manufacture, henceforth it will be necessary to inquire carefully into overall alignment accuracy. In other words, it is necessary to inquire minutely into each of such main factors as the alignment accuracy of the vertical SOR stepper itself, the strain (changes in position and dimension accuracy) of the X-ray photomask, the alignment accuracy between the X-ray photomasks, and the strain (curvature, deformation, etc.) of the wafer. It is important to carry out evaluation and inquiry in a form that includes overall alignment accuracy involving all these factors and the manufacture of the device.

#### 4.3 Device Manufacture

As mentioned at the beginning, SOR lithography has outstanding characteristics that are unobservable in other lithographies, not to mention its high resolution. It is now an important task to test manufacture devices that make the most of these characteristics. As mentioned above, studies have already been begun by IBM, NTT, West Germany, etc.

#### 4.4 X-Ray Photomask Technology

X-ray photomask technology is a theme that has been taken up most frequently as the subject of study since Professor Smith of MIT suggested X-ray lithography. However, when considering the X-ray photomask for SOR lithography use, there still remain important problems to be solved. In considering the near future, the SOR lithography to be put to practical use will probably be a transfer technology using the one-to-one close-exposure system (40-50  $\mu\text{m}$  gap between the photomask and the wafer). A major problem for realizing a resolution at the level near 0.1  $\mu\text{m}$  is posed by X-ray photomask technology. The author, however, considers that it is possible

to develop an X-ray photomask technology to satisfy these specifications, although its technical difficulty may possibly be significant. Hereafter, with the increase in expectations of the practical use of SOR lithography, it will become necessary to establish a high-precision X-ray photomask technology. The author considers it important in this case to pay attention to the following points. First, is to attach importance to the fundamental point of view. It is necessary to precisely evaluate and inquire into the position and dimension accuracy of the X-ray photomask. Research that digs a step deeper than conventional evaluation, that is, research from the scientific standpoint of physics and chemistry is necessary. For instance, such themes as the SOR irradiation resistance of the X-ray photomask membrane and the stress control of the W and Ta absorbers come under this category. Second is to select the X-ray absorber. The X-ray absorber is classified into two types--the conventional Au pattern by the plating method, and Ta and W pattern formed by the dry-etching method. In the former case, the pattern shape, stress, etc., can be relatively easily controlled, but it involves somewhat many processes and defect-related fears. The latter has a trend roughly contrary to the former. By the way, West Germany has finished basic studies up to the level of  $0.2 \mu\text{m}$  with regard to Au pattern-forming technology by the plating method.<sup>11</sup> IBM has used the Au pattern form test manufacturing devices.<sup>3</sup> Japanese photomask makers Dai Nippon Printing Co., Ltd., Toppan Printing Co., Ltd., and Hoya Corp. are grappling with the Ta pattern formation. After all, in the present situation, the final selection of technologies has not yet been decided. How the X-ray photomask technology level is filled up is the problem, but as mentioned also in the first point, it is believed that we may safely consider that we have not yet emerged from the basic stage. Consequently, it is considered that there is the need to steadily accumulate more basic data on the Au plating method and the Ta and W dry-etching method. At least, it is considered to be important to future progress to complete a thorough investigation and final elaboration of technology with regard to the Au plating method. Third is relative to the shape of the absorber pattern. Earlier, it had been regarded as optimal that the absorber pattern be rectangular. In recent research, however, it has been reported that inclining the pattern edge would rather improve the photomask contrast.<sup>12</sup> In addition, it has been reported that a rather thin absorber, which is  $0.5 \mu\text{m}$  thick, would be optimal. With regard to the pattern shape, too, research conducted by going back again to the starting point is considered to be necessary.

#### 4.5 X-Ray Photoresist

By the advent of SOR, requirements for the sensitivity of the X-ray photoresist have been slackened, but when considering the practical use of SOR lithography, the development of X-ray photoresists is indispensable. They must have an  $0.1 \mu\text{m}$  level resolution and an adequate dry-etching resistance. In addition, they are required to have a high sensitivity of  $<100 \text{ mJ/cm}^2$ . Further development is necessary for both the positive and the negative photoresists. Further, it is important that the photoresists do not spread.

## 5. Conclusion

SOR lithography is being expected to fill the need for half-submicron linewidth range pattern batch transfer technology for mass production. It is being especially regarded as most promising as a  $\leq 0.25 \mu\text{m}$  level transfer technology. At present, studies are being pushed in various places on such research and development themes as a subminiature SOR light source, a beam line exclusively for lithography, a vertical SOR stepper, an X-ray photomask, and an X-ray photoresist, but there are also many problems to be solved before their practical use. This paper has reported on the atmospheric exposure system SOR lithography technology that NEC has developed heretofore. It now is only at the stage of the foundation of the system having been solidified, and the company intends to push further research and development hereafter toward its practical use. The author expects that 1989 might possibly be a year when we can find a good prospect to develop a subminiature SOR light source, and wishes to plan, provided that it can come true, a new development of R&D strategy with this as a turning point of great importance for promoting SOR lithography research.

## References

1. A. Heuberger, J. VAC. SCI. TECHNOL., Vol B6, No 1, 1988, p 107.
2. K. Okada, Ibid., Vol A6, No 3, 1988, p 1611.
3. J.P. Silverman, V. DiMilia, D. Katcoff, K. Kwietniak, D. Seeger, L.K. Wang, J.M. Warlaumont, A.D. Wilson, and D. Crockatt, R. Devenuto, B. Hill, L.C. Hsia, and R. Rippstein, Ibid., Vol B6, No 6, 1988, p 2147.
4. NIKKEI MICRODEVICES, August 1988, p 66.
5. For instance, T. Tomiamasu, JPN. J. APPL. PHYS., Vol 26, 1987, p 741.
6. K. Okada, K. Fujii, Y. Kawase, and M. Nagano, J. VAC. SCI. TECHNOL., Vol B6, No 1, 1988, p 191.
7. E. Cullmann and K. Cooper, Ibid., Vol B6, No 6, 1988, p 2132.
8. K. Fujii, K. Okada, M. Nagano, and H. Kuroda, Ibid., Vol B6, No 6, 1988, p 2128.
9. E. Nomura and K. Okada, DIGEST 1ST MICROPROCESS CONF., 1988, p 90.
10. E. Kouno, Y. Tanaka, J. Iwata, Y. Tasaki, E. Kakimoto, K. Okada, K. Suzuki, K. Fujii, and E. Nomura [as published].
11. H.L. Huber, S. Pongratz, J. Trube, W. Windbracke, U. Mescheder, and F. Mund, J. VAC. SCI. TECHNOL., Vol B6, No 6, 1988, p 2184.
12. S. Hasegawa, J. Itoh, and N. Atoda, MICROELECTRONICS ENGINEERING (to be published).

**Basic Research by Research Reactor**

43062022 Tokyo PUROMETEUSU in Japanese Dec 88 pp 66-69

[Article by Atomic Energy Development Organization Controller's Office, Atomic Energy Bureau, Science and Technology Agency: "Atomic Energy--Hightech Groundwork Atomic Energy Research (11): Status of Basic Research by Research Reactor"]

**[Excerpt] 1. Status of Research Reactors**

It is about 30 years since the JRR-2, the first full-scale general-purpose test reactor in Japan, became critical in 1960. Since then, research using research reactors has been conducted continuously and the extent of this research and the number of researchers involved in it have continued to increase. Research reactors include single-purpose reactors, such as NSRR, whose sole purpose is the study of safety, but here, we shall discuss general-purpose reactors with relatively large outputs.

With its long history, the research reactor is now fairly well established in terms of safety technology but has still to be drastically improved in terms of experimental technology. The performance of research reactors has begun to be improved worldwide through renovation or remodeling to meet the rigorous needs of researchers. In Japan, JRR-3 of the Japan Atomic Energy Research Institute (JAERI) is being extensively remodeled to enable it to perform world-class research.

**2. Areas of Research Using Research Reactors**

The research reactor is used for the research and development of nuclear reactors through such uses as the irradiation of fuels and materials. The reactor is also used for conducting various types of basic research which utilize neutrons contained in the reactor. Its applications are, indeed, diverse but here, we shall discuss the state of the most recent basic research.

**Neutron Scattering and Diffraction**

The study of physical properties by neutron scattering and diffraction is the most active and the most productive of all applications of neutrons. In this experiment, precise measurement is made of the change of energy and

momentum of neutrons emanating from the sample, the object of measurement, as it is irradiated with neutrons extracted from the reactor. In this way, atomic arrangement in the sample and the strength of atomic bonding can be determined. Regarding the now much-noticed research on high-temperature superconductive substances, for instance, each researcher, in the initial stage, searched broadly for a new substance at his own discretion. This effort was sufficiently rewarded but few substances thus discovered have an established atomic structure. Also, no definite answer has been given to the question of why the phenomenon of high-temperature superconductivity occurs. Neutron scattering and diffraction experiments are a means suitable for this analysis of atomic structures. If, with the future advance in research, atomic structures are determined and the mechanism of high-temperature superconductivity is clarified, then substances with target properties may be produced by the reverse process of designing atomic structures with superior properties.

Neutron scattering and diffraction experiments are useful not only in the area of high-temperature superconductive substances. Research on a great variety of objects has already been conducted, in particular, research on the magnetic properties of metals. Furthermore, the latest tendency is for research to include not only metals, as in the past, but also substances with more complex or larger structures, such as polymers and biopolymers. Research on these objects requires measurements with a higher resolution. To this end, cold neutrons with a lower energy level than the thermal neutrons used in the past are preferable. These are expected to contribute much toward research on new materials, such as biotechnological materials and function polymers. Cold neutron sources are being extensively established world-wide since cold neutrons have superior properties for other areas of basic research, as well. In the remodeling of JRR-3, the establishment of cold neutron sources is planned.

#### Basic Physics

In the area of basic physics, neutron physics is closely related to elementary particle physics which is based on high energy accelerator. Such items as the electric dipole efficiency, the life of neutrons and the presence of electric charge are measured using an nuclear reactor. Its progress is remarkable in terms of experimental technology, including the capability of generating super-cold neutrons with a velocity of about 5 m/s and containing them in a hollow metal bolt.

#### Neutron Radiography

Neutrons are not used only for basic research. Typical of their industrial applications is neutron radiography which, unlike X-rays, is a useful means of nondestructive inspection necessary for light elements, such as hydrogen. Its pictures have recently improved qualitatively. Also, it can now detect changes by being interlocked with a television system. In fact, there is a television commercial message showing pictures of oil movement in an engine. Moreover, computer tomography using neutrons has been developed.

## Neutron Irradiation

The above are neutron beam tests but another major area using a research reactor is neutron irradiation. Activation analysis has already been established as a method of trace element analysis and its applications range widely from archeology to the study of environmental pollution. Its industrial applications include the production of semiconductor materials by silicon doping and the evaluation of effects of neutron irradiation of nuclear reactor materials. As new technologies, research is being conducted on trace element analysis using prompt  $\gamma$  rays that can measure elements different from those for the conventional activation analysis, the use of heat pipes for efficient heat elimination in neutron irradiation with a high exothermic density and the use of shape memory alloys under radiation.

### 3. Role of Research Reactor

As stated, research using the research reactor is diverse. Experiments in the neutron irradiation of fuels and materials is obviously important because there is no alternative. In experiments on neutron scatter and diffraction, meanwhile, new technologies have been introduced and neutrons from the nuclear reactor are expected to be used in the future, in a way to make the most of their characteristics.

Aside from neutron tests, the use of synchrotron radiation is now noted most as a means of structural analysis at the atomic level. But whereas the scattering cross-sectional area of atomic nuclei for neutrons is extraordinarily large with hydrogen regardless of atomic numbers, in synchrotron radiation the contribution from elements with small atomic numbers is small because it is scattered mainly by electrons. It is, therefore, better to use neutrons than synchrotron radiation for the study of hydrogen and other light elements. Neutrons are better for the study of biopolymers, for example, because hydrogen has an important flat-panel display in them. Furthermore, whereas biological samples used in structural analysis using synchrotron radiation or X-rays require crystallization or some other special solidification, samples used in neutron tests do not require it and thus tests can be conducted under conditions closer to the organism. Neutron tests are also suitable for the study of magnetic substances because neutrons have magnetic moments. Therefore, tests using neutrons bring information that cannot be obtained from tests using synchrotron radiation.

As neutron sources of great strength, there is the continuous neutron source using the nuclear reactor and the pulse neutron source using the accelerator. The pulse neutron source has many merits including its peak neutron flux which is higher than the peak neutron flux by the nuclear reactor but the pulse width increases and the number of peak neutron fluxes decreases if the neutrons are decelerated to obtain low-energy neutrons. Meanwhile, the nuclear reactor, which is the continuous neutron source, has the advantage of a high time-average neutron flux. So, it is convenient for generating neutrons with low energy from cold neutrons down. Thus, the nuclear reactor can be flexible for the needs of different energy areas according to different tests.

#### 4. Outlook

The remodeling of JRR-3 long awaited by researchers is making steady progress. Since it can use high neutron fluxes, it will make possible unprecedented research. As indicated in Photo 1 [not reproduced] and Figure 1, the number of test devices will sharply increase. So, diverse devices will be planned to conduct research in many areas and many studies will become possible. Further, as indicated in the system diagram in Figure 2, a cold neutron source will be installed, making it possible to use low-energy neutrons. Thus, research in new areas including biopolymers will advance. This cold neutron source is designed to produce liquid hydrogen of about 20 K, using helium freezing equipment, lower the energy of neutrons passing through the moderator container containing this liquid hydrogen and provide it for tests. When this remodeling of JRR-3 is completed, it is expected to greatly improve neutron research both in quality and quantity. Figure 3 outlines specific research trends.

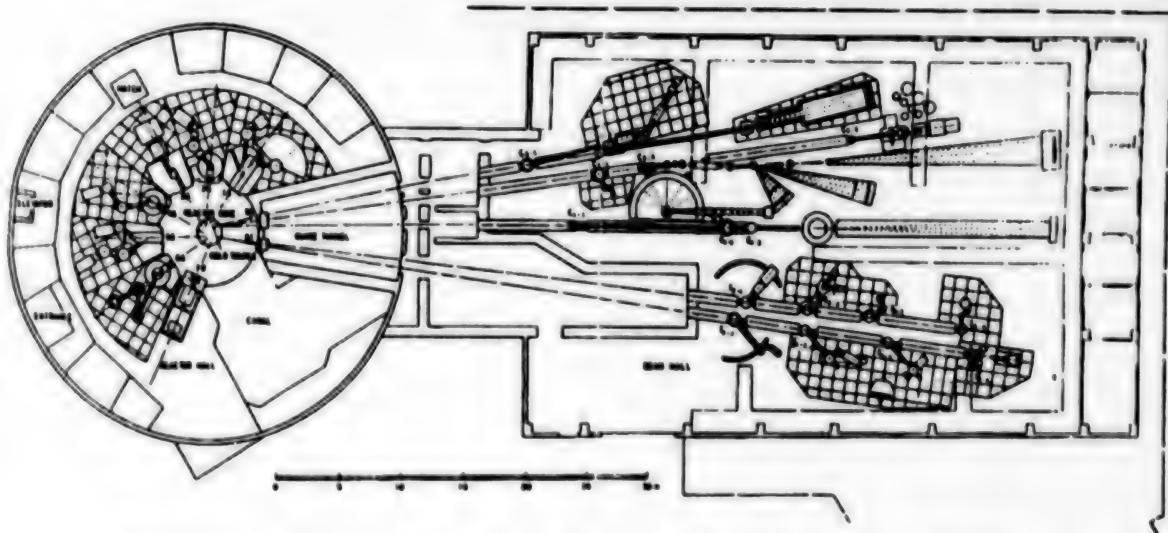


Figure 1. General Plot Plan of Neutron Beam Testing System

Beam testing system in  
nuclear reactor building

Beam testing system using neutron conduits

No	Name of apparatus	No	Name of apparatus	No	Name of apparatus
1G	High-resolution powder neutron diffractor	T1-1	High resolution triaxial spectrometer (university)	C1-1	High-energy resolution triaxial spectrometer (university)
2G	Triaxial neutron spectrometer	T1-2	Neutron diffractor (university)	C1-2	Two-dimensional position measurement small-angle scatterer (univ.)
3G	Neutron topography	T1-3	Powder neutron diffractor (university)	C1-3	Super-high resolution rear scatter (univ.)
4G	Universal triaxial neutron spectrometer (university)	T1-4	Prompt $\gamma$ -ray analyzer	C2-1	Low-energy triaxial spectrometer
5G	High-performance polarized neutron scatterer (university)	T1-4	Neutron camera diffractor (university)	C2-2	Neutron spectrum modulator (university)
		T2-1	Neutron diffracting and picturing device	C2-3	Neutron radiography, prompt $\gamma$ -ray analyzer
6G	Neutron spectrometer (university)	T2-2	Neutron interferometer (university)	C2-3	Ultra-cold neutron generator (university)
7R	Neutron radiography	T2-3		C3-2	High-resolution neutron spin echo device (university)
		T2-4	High-resolution triaxial neutron spectrometer	C3-2	Neutron small-angle scatterer

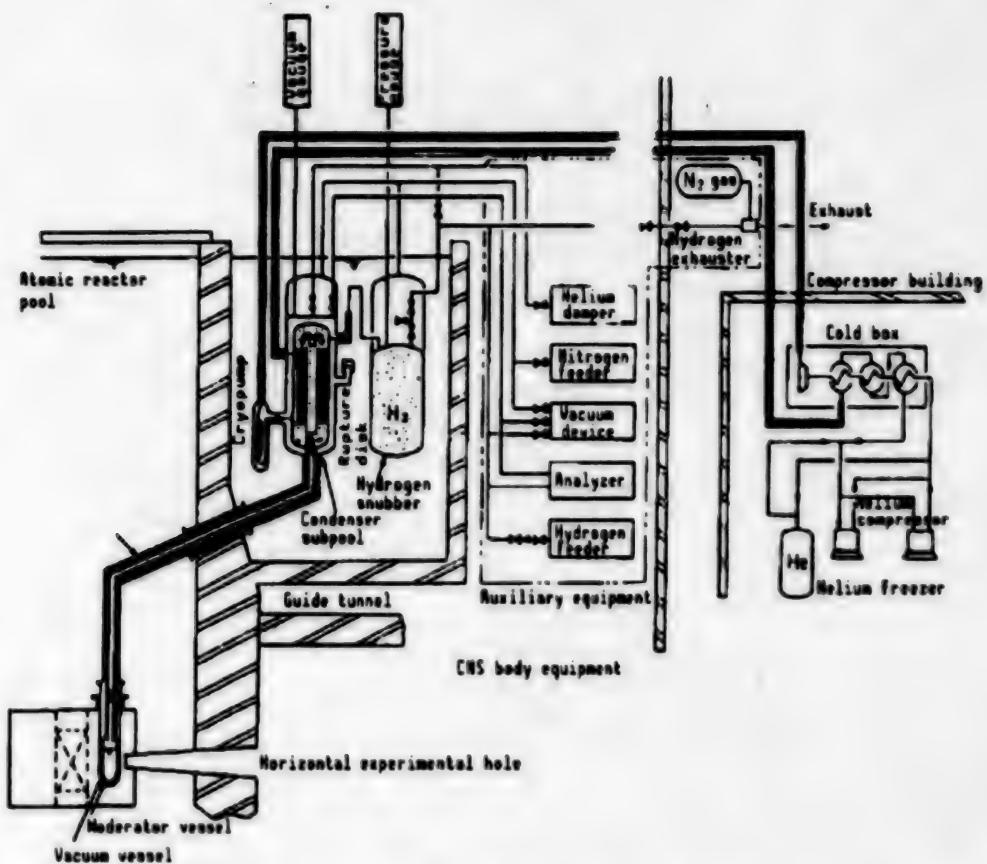


Figure 2. Drawing of JRR-3 Cold Neutron Source Equipment System

It is not too much to say that Europe--notably the Center for Nuclear Research in Grenoble, France--now leads the world in neutron research.

As in high-temperature superconductivity research, it is important that the different areas of most advanced research should be closely interrelated for prompt mutual response. It is hoped that the research reactor will continue to be used in the future by researchers in many areas.

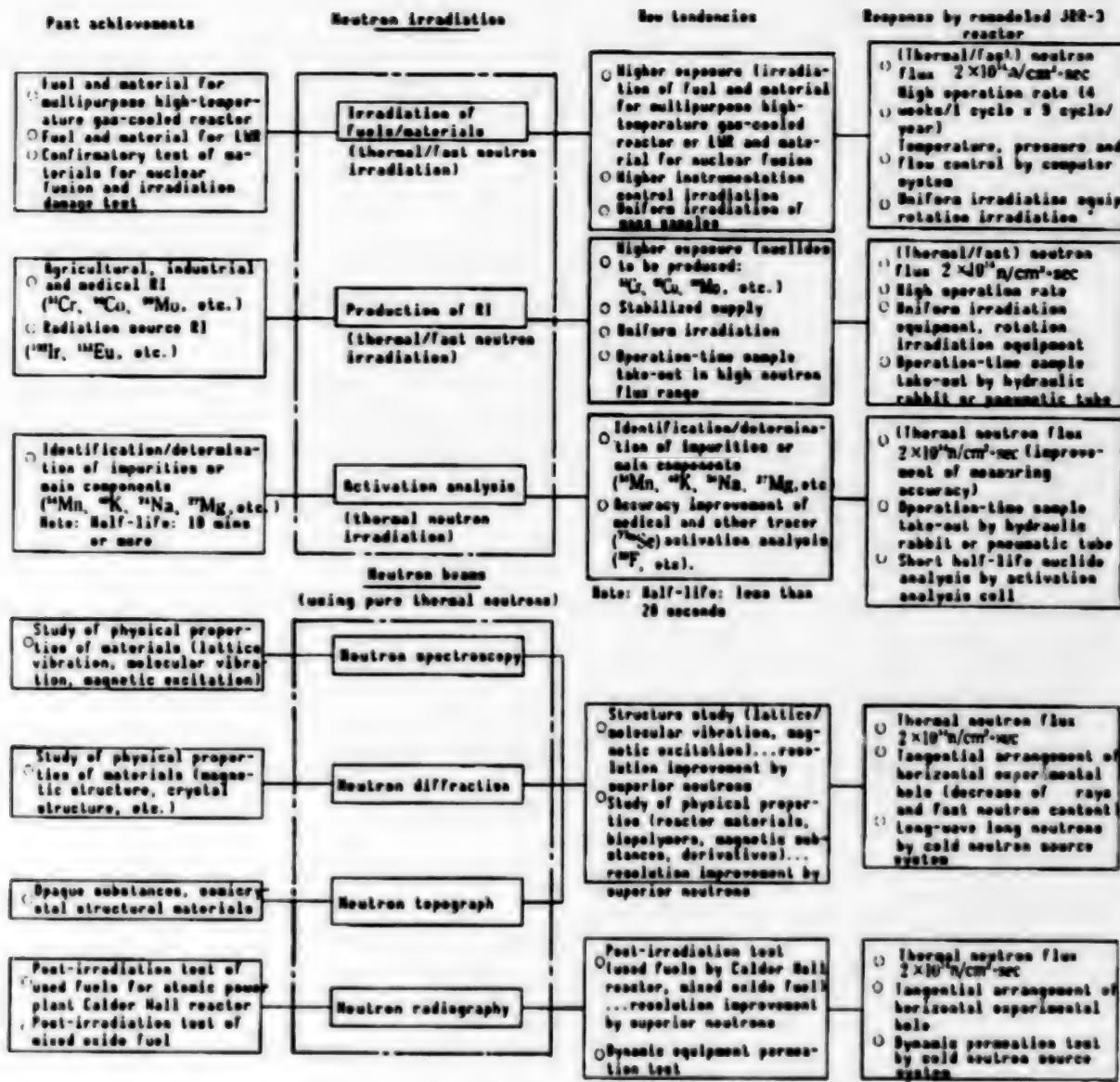


Figure 3. Trends in the Use of Research Reactors

## NUCLEAR ENGINEERING

### Nuclear Fusion Reactor Core Research Status Examined

43063520 Tokyo PUROMETEUSU in Japanese Feb 89 pp 60-63

[Article by Technology Promotion Section, Atomic Energy Bureau, Science and Technology Agency]

[Text] Since the resources for the fuel used for nuclear fusion are abundant; it is expected that a great volume of energy can be obtained from nuclear fusion. The technological development of nuclear fusion which is considered to play the role of leading other advanced technologies, is, from the long-term point of view, an important task for Japan, which lacks in energy resources, to tackle positively.

#### I. Introduction

Japan's nuclear fusion R&D currently underway is based on "the basic plan for second-phase nuclear fusion R&D" (hereafter referred to as "the second-phase plan") worked out by the Atomic Energy Commission in 1975 and the "atomic energy development and utilization long-term plan" (hereafter referred to as "the long-term plan") worked out by the same commission in 1987.

The second-phase plan is mainly comprised of two R&D projects: (1) nuclear fusion reactor core plasma technology and (2) nuclear fusion reactor core engineering technology and nuclear fusion reactor engineering technology. The long-term plan, which positions nuclear fusion R&D as a leading project, sets out a basic direction for the R&D activities after the targets of the second-phase plan have been achieved. The critical "JT-60" plasma-testing equipment of the Japan Atomic Energy Research Institute, which plays an important role in the second-phase plan, has reached the targeted region of critical plasma conditions provided by the Atomic Energy Commission, ascertaining scientifically the prospects for the practical use of nuclear fusion (Figure 1). According to the long-term plan, the next goal is to "achieve self-ignition conditions and combustion for hours around the year 2000 and at the same time to demonstrate basic reactor engineering technology. Viewed from the present stage of research, this goal is expected to be attained through the nuclear fusion reaction of heavy

hydrogen (D) and tritium (T). Therefore, R&D aimed at making the next large-scale device for this purpose more efficient and optimum is now being positively conducted.

Out of the R&D activities that concern the next large-scale device and are essential for the creation of a nuclear fusion reactor, the R&D on reactor core plasma being conducted by the Japan Atomic Energy Research Institute will be introduced.

## II. Research on reactor Core Plasma

### 1. High Efficient Confinement

Although D-T reaction is the best in generating nuclear fusion reaction, simulations of D-T reaction conditions (temperature, density, energy confinement time, etc.) are being conducted by using hydrogen and heavy hydrogen or helium at present. This is intended to see if by the use of hydrogen, etc. one can achieve the conditions under which D-T reaction ensures stable combustion, self-ignition, and continued combustion, producing high temperatures and high density and confining plasma energy efficiently. In order to generate the condition for self-ignition it is necessary to confine the energy of plasma of a high temperature and high density for over a certain period of time (1-2 seconds). High temperatures can be obtained by increasing additional heat input, while high density can easily be obtained by injecting fuel rapidly in the form of a small ice flake. However, it is not easy to confine the energy of plasma of a high temperature and high density for a specified period. As additional heat input is increased to produce a higher temperature, the energy of plasma is liable to escape. On the other hand, since the energy-confinement time is proportional to the electric current of plasma, in order to increase the plasma current it is necessary to increase the cross section of plasma, which in turn calls for a device of a larger scale.

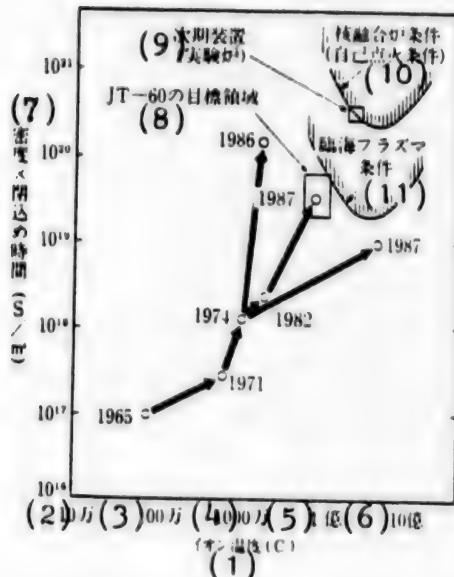


Figure 1. Evolution of Tokamak's Confinement Performance

Key:

1. Ion temperature (°C)	7. Density X confinement time (S/m³)
2. 100,000	8. Target region of JT-60
3. 1 million	9. Next device (experimental reactor)
4. 10 million	10. Nuclear fusion reactor conditions
5. 100 million	(self-ignition conditions)
6. 1 billion	11. Critical plasma conditions

The performance values of Tokamak devices in the world are shown by year. The frame near the center indicates the target region for JT-60 set by the Atomic Energy Commission. For critical plasma, lines are shown considering only nuclear fusion by thermalized plasma. For achieved plasma, temperature density central values are used.

For adding heat there are two methods. One is to inject a great amount of atoms into plasma from the outside and make them collide against each other (neutral particles injection heating, Figure 2). The other is to put ions contained in plasma or electrons, or both of them, on the high frequency provided from outside, and make them resonate (high frequency heating, Figure 3). A phenomenon (L mode) that additional heating, if made further, lowered the efficiency of plasma energy confinement, has heretofore been observed. However, despite additional heating, the same energy-confinement efficiency (H mode) as before additional heating was obtained around 1982 by using the device,

ASDEX-111, of the Max Plank Plasma Physics Research Institute of West Germany. Afterward, the H mode was also confirmed by the D-111 device, under cooperation between Japan and the United States, but found not lasting long. However, recent experiments by the D-111D have produced a long-lived H mode. On the other hand, it has been discovered by JFT-2M of JAERI that the H mode shifts to a new more efficient mode in confinement (Fig. 4). Furthermore, a new mode that improves the L mode by about 50 percent by the intensive input of additional heat has been discovered by JT-60. Discussions are now being conducted on the mechanism involved here from theoretical and experimental viewpoints.

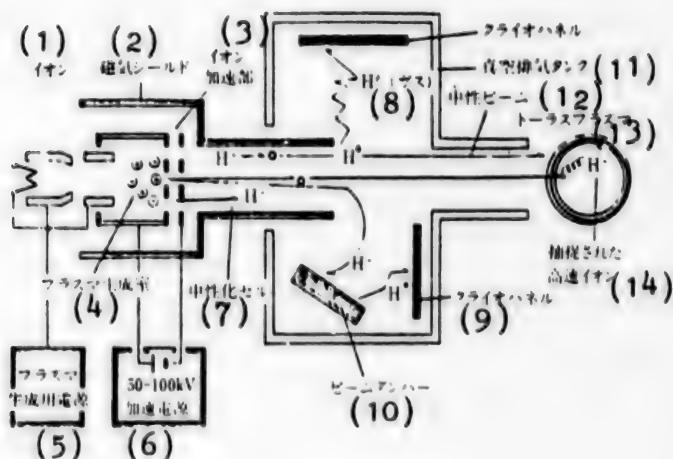
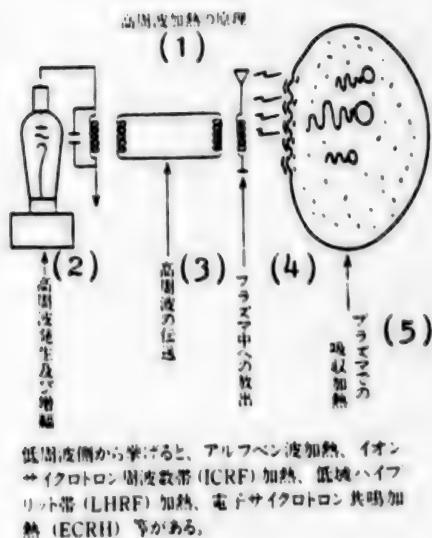


Figure 2. Neutral Particles Injection (NB1) Heating

Key:

1. Ion
2. Magnetic shield
3. Part of ion acceleration
4. Plasma production chamber
5. Power source for plasma production
6. Power source of 50-100 kV for acceleration
7. Neutralized cell
8. Gas
9. Cryopanel
10. Beam damper
11. Vacuum exhaust tank
12. Neutral beam
13. Torus plasma
14. Captured fast ion



低周波側から挙げると、アルフヘン波加熱、イオン  
サイクロotron周波数帯 (ICRF) 加熱、低域ハイブ  
リット帯 (LHRF) 加熱、電子サイクロotron共鳴加  
熱 (ECRH) である。

Figure 3. Radiofrequency Heating

Key:

1. Theory of radiofrequency heating
2. Generation and amplification of high frequency
3. Transmission of high frequency
4. Discharge into plasma
5. Absorbing and heating in plasma

Citing from low frequency, there are Alfven wave heating, ion cyclotron frequency band heating (ICRF), a low-region hybrid band (LHRF) heating, and electron cyclotron resonance heating (ECRH).

The improvement of energy confinement efficiency is an important element of research, since it ensures the self-ignition of a nuclear fusion reactor and also leads to the scaling down of the reactor.

2. Steady Operation of Tokamak Furnace

Tokamak is to apply an electric current to annular (torus shape) plasma and confine it in the magnetic field thus created. However, it is "evolving" from the existing induction current method, which generates an electric current through changes in pulsed flux changes, to the current drive method, which provides speed in a specified direction to the electrons or ions in plasma by additional heating. An electric

current is driven while inputting additional heat, and plasma is confined in the magnetic field thus created. And, additional heating for many hours can result in the steady operation of the tokamak furnace.

JT-60 has achieved continuous operation for about 3 seconds with the high frequency of a low-region hybrid wave band by driving a current of 2MA and TRIAM-1M of Kyushu University has also achieved it for 3 minutes 12 seconds by driving a plasma current as small as 28KA.

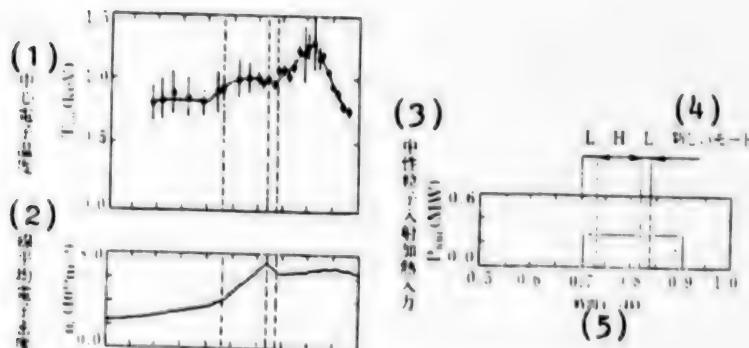


Figure 4. New Confinement Mode

Key:

1. Central electron temperature
2. Linear electron density average
3. Injection, heating and inputting of neutral particles
4. New mode
5. Time  $t$  (second)

Moreover, the D-111-D has driven a plasma current of 340KA by injecting neutral particles. It is a task to successfully drive electric currents in plasma of higher density in order to introduce these results into a next large scale device.

### 3. Relationship between Plasma and Wall Surface

An important element, like the condition for additional heating, that affects the efficiency of plasma energy confinement is the state of a vacuum container's internal wall (called the first wall), which faces the plasma. Since impurities (an atom of a great valency) getting mixed into plasma cause plasma energy to be lost through radioisotopes, resulting in the decay of plasma, it is necessary to lessen impurities in plasma.

Traditionally, internal wall material has been prevented as much as possible from melting into the plasma by bringing the outer circumference of the plasma into contact with the projections (limiter) inside the vacuum container while keeping it from touching the container's internal surface. Thus in 1973 at the time JAERI's JFT-2a was being designed, the approach (magnetic limiter approach/diverter approach) was devised in which, by keeping the perimeter of the plasma from contact with the inner wall of the vacuum chamber, and causing the impurities which "vaporize" from the inner wall and which tend to flow into the plasma to intersect with the magnetic lines of force on the perimeter, they (the impurities) are guided outside the plasma.

The results of this method were reflected on JT-60 and this method is essential for a future nuclear fusion device as seen from the fact that JFT, after the start of operation, has improved confinement efficiency by adopting the magnetic limiter method, which is incorporated in the conceptions of the next large-scale devices of various kinds.

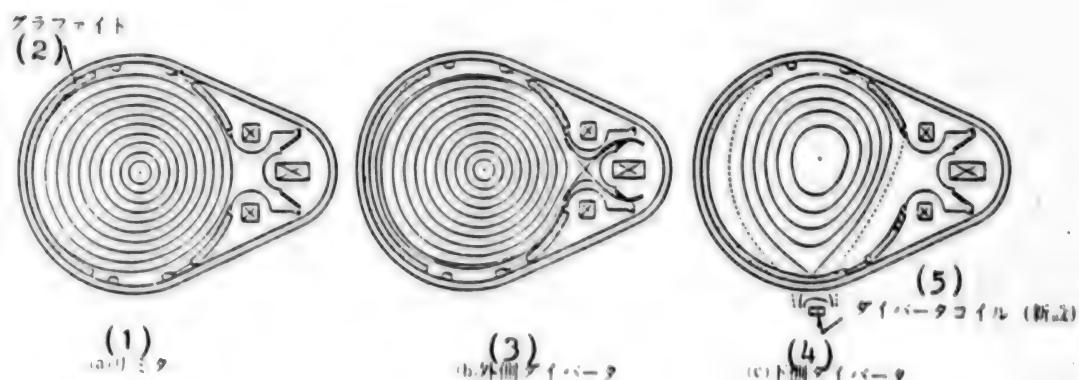


Figure 5. Arrangement of Diverter of JT-60

Key:

1. Limiter	4. Lower diverter
2. Graphite	5. Diverter coil (newly installed)
3. Outer diverter	

The selection of the material of the inner wall of the vacuum container, which faces the plasma, is highly important. It must be of such a quality that even if mixed with plasma, it does not cool plasma, withstands high temperatures and is not damaged by fast neutrons.

#### 4. Prevention of Decay of Plasma

During the production of plasma, it suddenly decays sometimes if something is unstable. This is called disruption. At the time of disruption, its energy collides against the first wall intensively and digs into the wall, and the device vibrates.

This phenomenon occurs when the density of plasma exceeds certain limitations, impurities get mixed in plasma and instability is generated.

JT-60 prevents disruption by gradually terminating plasma discharge under the condition of additional heat being injected. However, more basic preventive measures are being sought.

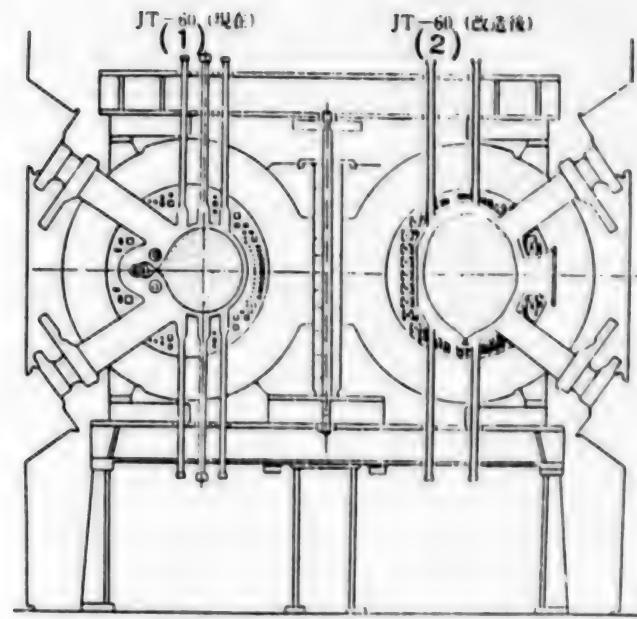
### III. Future Direction of Research

The research on nuclear fusion reactor core plasma is making rapid progress.

With respect to the tokamak device, the checking of theoretical analyses against experiment results are being made in expectation of realizing "high-efficiency confinement" that leads to a compact device, and "steady operation of high-density plasma."

JT-60 will be rebuilt during the period from 1989 to 1990, retaining most of the current facilities useful, in order to secure the data base related to the core plasma of the next large-scale device (Figure 6). JFT-2M, playing a forerunner's role in a pioneer study of core plasma, taking advantage of its mobility as a middle-scale device, will be conducting experiments to facilitate the gradual improvement of core plasma of a next device while improving its facility.

Experiment results through the combination of middle-scale and large-scale devices will lead the world and offer an important arm to the R&D of core plasma after the next device and thereafter.



(3) JT-60の主要諸元		(11) JT-60改修後 主要諸元	
(4) プラズマ主半径	(m) 3.0	4) プラズマ主半径	(m) 3.4
(5) プラズマ副半径	(m) 0.6	5) プラズマ副半径	(m) 1.0 × 1.3
(6) トロイダル磁場	(T) 4.3	6) トロイダル磁場	(T) 4.2
(7) プラズマ電流	(MA) 2.7	7) プラズマ電流	(MA) 6.0
(8) 同上断続時間	(sec) 10	8) 同上断続時間	(sec) 15
(9) 中性粒子入射加熱入力	(MW) 20	9) 中性粒子入射加熱入力	(MW) 40
(10) 高周波加熱入力	(MW) 10	10) 高周波加熱入力	(MW) 20
		(12) 高周波電流駆動入力	(MW) 20

Figure 6. Rebuilding of the JT-60 for Enlarging Electric Current

Key:

1. At present	9. Injection, heating, and input of neutral particles
2. After remodeling	10. High-frequency heating and input
3. Main specifications	11. Main items after rebuilding
4. Main plasma radius	12. High-frequency current drive input
5. Auxiliary plasma radius	
6. Toroidal field	
7. Plasma current	
8. Plasma current continuation time	

### Superconducting Oxide Fiber With Coating Film

43067085 Tokyo PROCEEDINGS OF NEW CARBON AND NEW CERAMICS FIBER CONFERENCE  
in Japanese 16 Dec 88 pp 30-36

[Article by S. Sakka of Kyoto University]

#### [Text] 1. Introduction

A round or ribbon-shaped conducting wire is needed for the practical application of high-temperature oxide superconductors. Various methods are used to make such a conducting wire. One is to form a superconductive film around a ribbon - shaped or wire-shaped basic body by coating, a second is to draw superconductive ceramics into the form of a wire and a third is to bundle superconductive ceramic fibers together to form a wire. This article will describe a process for manufacturing a coating film and fiber composed of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\sigma}$  by sol-gel processing. We succeeded only in producing a coating film that displayed superconductivity at a critical temperature not sufficiently high and a fiber that was mechanically brittle with a critical temperature that was not too high. We would feel happy, however, if our experiments contribute to future advancements in this field.

#### 2. High-Temperature Superconductors and Conventional Superconductors

A superconductor shows the Meissner effect--zero electric resistance--below a critical temperature( $T_c$ ) and it does not filter the line of magnetic force. These phenomena have a quantum effect that appears macroscopically as a result of electron-pairs. These pairs, called Cooper-pairs, follow Bose statistics, traveling through the substance without losing kinetic energy.

Superconductivity in mercury was discovered by Kamerlingh-Onnes in 1911 and was later found in metallic elements such as Pb, Nb and Tc, and in metal alloys such as  $\text{Nb}_3\text{Sn}$  and  $\text{Nb}_3\text{Ge}$ . Research on wiremaking was conducted using such alloys as  $\text{Nb}_3\text{Ge}$ . Between 1973 and the mid-1980s, superconductivity was discovered in such oxides as  $\text{Li}_2\text{TiO}_4$  and  $\text{Ba}(\text{Pb},\text{Bi})\text{O}_3$ . The fact that superconductivity exists not only in metals has directed researchers' attention to these oxides. However, since their  $T_c$  is about 13 K, which is

low compared with 18 K for  $\text{Nb}_3\text{Sn}$  and 23.6 K for  $\text{Nb}_3\text{Ge}$  metal alloys, only a few ceramics researchers, including Professor Hiroshi Kitazawa, took an interest in them as practical materials. In 1986 Bednorz and Muller reported that the  $T_c$  of  $(\text{La}_{1-x}\text{Ba}_x)_2\text{CuO}_4$  is as high as 30 K, and a group from Tokyo University revealed that the  $T_c$  of  $(\text{La}_{1-x}\text{Sr}_x)\text{CuO}_4$ , which is similar to a perovskite-type crystal, is about 40 K. Ever since the situation has changed. The exploration of oxides with higher  $T_c$ s and research on processing high-temperature superconductive ceramics and superconductivity theories explaining high critical temperatures have been actively pursued. Table 1 shows the kinds of high-temperature superconductive oxides.

With respect to conventional low-temperature superconductors--metallic elements and metal alloys--a curve was drawn indicating a border between a superconductive state and a normal conductive state, with  $T_c$  as a horizontal axis and a critical field( $H_c$ ), which does not break the superconductive state, as a vertical axis. It was shown that a superconductor with a high  $T_c$  also has a high  $H_c$ , and these curves did not cross. Moreover, since a homogeneous composition without pores is easily obtainable from metal alloys, their critical current density( $J_c$ ), which indicates the limit at which the superconductive state is broken into a normal conductive state with regard to electric currents, is high so that no serious problems have arisen. While high-temperature superconductive ceramics have a defect in that their  $T_c$  is high, their  $J_c$  is generally lower than those of metal alloys, as shown in Figure 1. Additionally, the  $T_c$  sometimes does not reach the value, which seems to be the highest value for its substance, according to its manufacturing process. This is because high-temperature superconductive ceramics are generally polycrystal and do not become a crystalline seed that displays superconductivity if a sufficient amount of oxygen does not enter its structure, as shown in Table 2, which takes  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\sigma}$ , for example. Thus, merely producing a dense ceramic sinter does not solve the problem. This means that in order to produce excellent superconductive ceramics it is necessary that the superconductive phase, which contains a lot of oxygen, be densely sintered (crystalline particles firmly bonded with surrounding crystal particles so that there are no gaps or pores between them). However, dense sintering makes it difficult for oxygen to enter, resulting in a crystal phase with a low  $T_c$ , while a porous condition that allows oxygen to enter freely provides only small contact areas between particles, resulting in a low  $T_c$  and a low  $J_c$ .

Such problems must be taken into consideration, as a matter of course, in manufacturing high-temperature superconductors by the sol-gel processing method, which will be described in this article.

### 3. Manufacturing High-Temperature Superconductors by Sol-Gel Process

Before turning to coating films and fibers, we will examine the application of the sol-gel process to the manufacture of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  ceramics.

Table 1. High-Temperature Superconductive Ceramics

Composition	Tc (K)	Year	Discoverer
$(La_{1-x}Ba_x)_2CuO_{4-\sigma}$	30	1986	Bednorz Muller
$(La_{0.32}Sr_{0.08})_2CuO_{4-\sigma}$	37	1986	Tanaka, et al.
$YBa_2Cu_3O_{7-\sigma}$	94	1987	Chub
$BiSrCaCu_2O_x$	104	1988	Maeda et al.
$Tl_2Ba_2Ca_1Cu_2O_{8+8}$	100	1988	Sheng Hermann

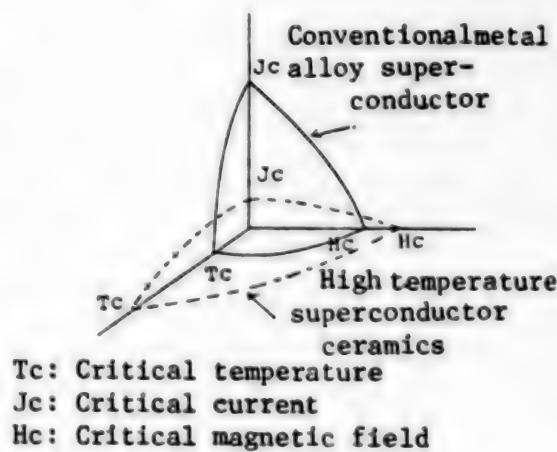


Figure 1. Three Kinds of Superconducting Critical Values

Table 2. Problems of High-Temperature Superconductive Ceramics Composed of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\sigma}$

<u>Problem</u>	<u>Explanation</u>
Generation of homogeneous superconductive single phase	Superconductive phase is generated
Existence of phase transition	Since the superconductive phase stabilizes at a low temperature, sintering at a high temperature is not proper.
Oxidization of $\text{Cu}^{2+}$ to $\text{Cu}^{3+}$ and introduction of oxygen	If it is densely sintered, oxygen can barely penetrate.
Sintering to ensure a dense structure	Since the superconductive phase disappears at high temperatures, it is difficult to sinter.
Development of pores	Pores are apt to develop due to oxygen moving in and out.

The sol-gel process<sup>1</sup> begins with solutions of metallic compounds (metallic alkoxide, acetylacetone, metallic fatty acid chloride and metallic nitrate, etc.), which are bases for objective metallic oxides. Compounds undergo solation in solutions through hydrolyses and polymerization reactions, and solidify as a gel as the reactions progress further. The gels are then heated to produce oxides. In general, characteristics of this process make it possible to produce dense ceramic sinters at low temperatures. Since solutions can easily be formed into fibers and coating films before gelling, they can be used to make plastic bodies other than bulk plates and rods. These characteristics are demonstrated to some extent when the sol-gel process is applied to a superconductor composed of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\sigma}$ <sup>2</sup>. It will be shown in this chapter that the sol-gel process produces compounds at lower temperatures than the conventional process that uses mixtures of carbonate and oxide powder.<sup>3</sup>

The author and his colleagues used acetates as the starting compound for the sol-gel process. As shown in Figure 2, acetates of Y, Ba and Cu are melted in distilled water, an ammonia solution is added, and then the PH is adjusted to 6-6.1. A transparent blue solution is thus obtained. If it is enriched at 60-80°C until its volume is reduced to about one-tenth that at the time of preparation, it becomes a viscose transparent blue sol. If made into a solid transparent blue gel through further reactions and then heated for 5 hours at 910°C in air, it becomes a superconductive ceramic with a  $T_c$  (the temperature at which electric resistance becomes zero) of 90 K, as shown in Figure 3. However, when its temperature reaches 200-300°C, it begins to foam and pores are created. Even after being heated at 910°C the pores do not disappear, and thus the product has a porous appearance.

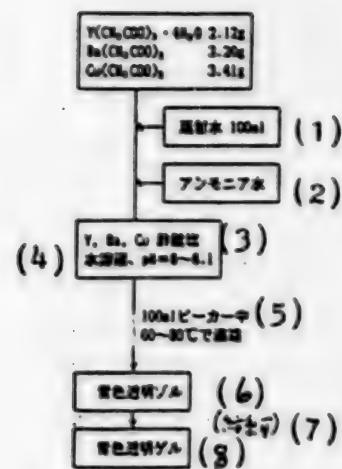


Figure 2. Manufacture of Gel From Acetate Solution

Key:

1. Distilled water	5. Enrich at 60-80°C in a 100 ml beaker
2. Y, Ba, Cu ammonia solution	6. Transparent blue sol
3. Acetate	7. (Can be spun)
4. Solution, pH = 6-6.1	8. Transparent blue gel

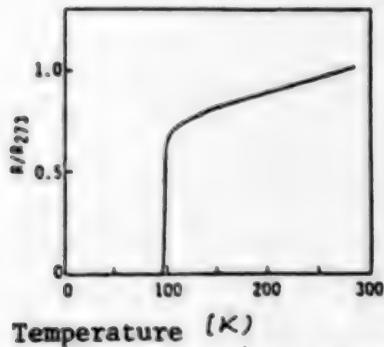


Figure 3. Resistance-Temperature Curve of Gel Sinter  
After calcining for 5 hours at 910°C, it was cooled in the furnace.

During the heating process, various compounds are produced and disappear. When the gel produced by the sol-gel process was heated, a  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\sigma}$  superconductive oxide or other similar compounds were produced at 750°C, as can be seen in the diffraction chart in Figure 4. Although other oxides providing small diffraction peaks remained, the large peaks represent superconductive oxides. When powdery raw materials mixed in a milk bowl were heated to 750°C using the same procedure, superconductive oxides did not separate out. This indicates that the sol-gel process is also a low-temperature synthesizing process with regard to superconductive oxides.

#### 4. Manufacture of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\sigma}$ Coating Film by Sol-Gel Process

In general, with metallic alkoxyl as a starting compound, the sol-gel process primarily uses solvents of the water-alcohol system.<sup>1</sup> This is because, if metallic alkoxyl group (OR group such as  $\text{OCH}_3$  and  $\text{OC}_2\text{H}_5$ ) becomes a hydroxyl group (OH) through hydrolysis, and this part is likely to create a metal-oxygen-metal bond by releasing  $\text{H}_2\text{O}$  when acting on an OH radical of another alkoxide molecule that has received hydrolysis, and it becomes a metallic oxide particle through the advancement of polymerization.

However, an alkoxide of Cu does not dissolve in water-alcohol solvents. Therefore, realizing that ethanolamines easily dissolve Cu compounds, we used a liquid mixture of methanol and triethanolamine as a solvent.<sup>4</sup> As shown in Figure 5, metallic barium (Ba) was dissolved in methanol to produce a methanol solution of  $\text{Ba}(\text{OCH}_3)_2$ ; a solution of  $\text{Y}(\text{n-OC}_4\text{H}_9)_3$ , which was previously dissolved in a solution of methanol-triethanolamine, was added; and  $\text{Cu}(\text{OCH}_3)_2$  was then added to the solution thus prepared and dissolved in it to prepare a homogeneous coating solution. This coating solution was dripped on to a substrate of stabilized zirconium oxide (the zirconium oxide is stabilized to prevent a  $\text{ZrO}_2$  cubic crystal from crystal transition by adding  $\text{Y}_2\text{O}_3$  which acts a little upon superconductive compounds while firing). It is then dried and heated at 800°C for 5 minutes. After repeating the cycle of dripping-drying-heating 5-10 times to get a thicker film, the solution was heated in oxygen for 60-80 hours at 800°C. A superconductive ceramic coating film with a thickness of about 7  $\mu\text{m}$  was thus obtained. Heating in  $\text{O}_2$  is a process that can be applied in case superconductive properties do not occur due to insufficient oxygen in the crystal structure. This is called annealing.

As a photo taken by a scanning electron microscope shows (Figure 6) a superconductive film with a thickness of several microns consisting of about 1  $\mu\text{m}$  particles can be obtained. This film displays a superconductive property with a  $T_{\text{c}}(\text{end})$  of about 60 K, as shown in Figure 7.

There is a process for making a  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\sigma}$  superconductive coating film that uses solutions of high fatty acid salts like metallic stearic acid salts, naphthenic acid salts and acetates. This is in addition to the sol-gel process, which uses metallic alkoxide.

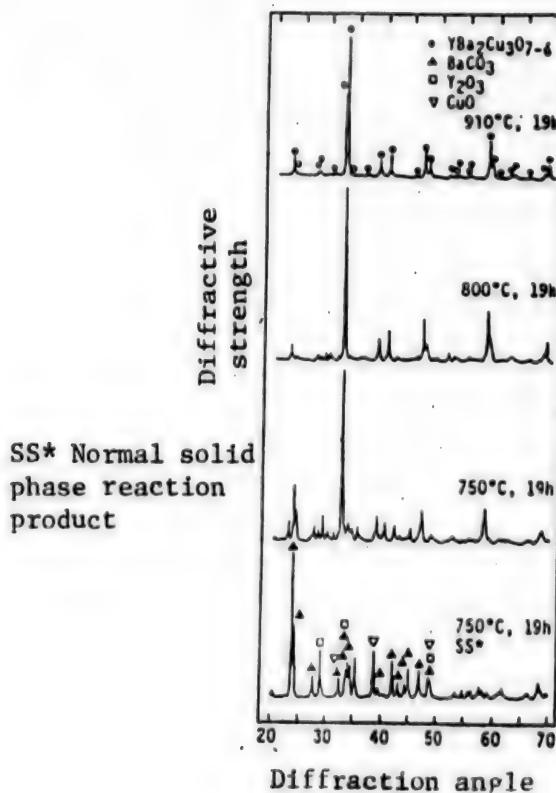


Figure 4. X-Ray Diffraction Pattern of Gel Sinter

In any case, since a solution is heated at a high temperature of 800-900°C to make a film superconductive, reactions between the substrate and the ingredients of the film are unavoidable. Acting upon the substrate, Ba ingredients in particular penetrate the substrate selectively and, as a result, Ba ingredients in the film become short. Thus, in the part of the film near the substrate, in the direction of the thickness, nonsuperconductive compounds are produced. To prevent this, it is necessary to ensure that the thickness of the film is increased by repeating the coating process to precipitate only superconductive  $YBa_2Cu_3O_{7-\delta}$  in the part of the film far from the substrate.

##### 5. Manufacture of $YBa_2Cu_3O_{7-\delta}$ Fiber by Sol-Gel Process

The first step is to search for a solution from which spinning can be done, so that fibers can be manufactured by the sol-gel process. When the viscosity of the solution is increased through reaction, it is spun to produce gel fibers, which are then heated to develop superconductive ceramic fibers.

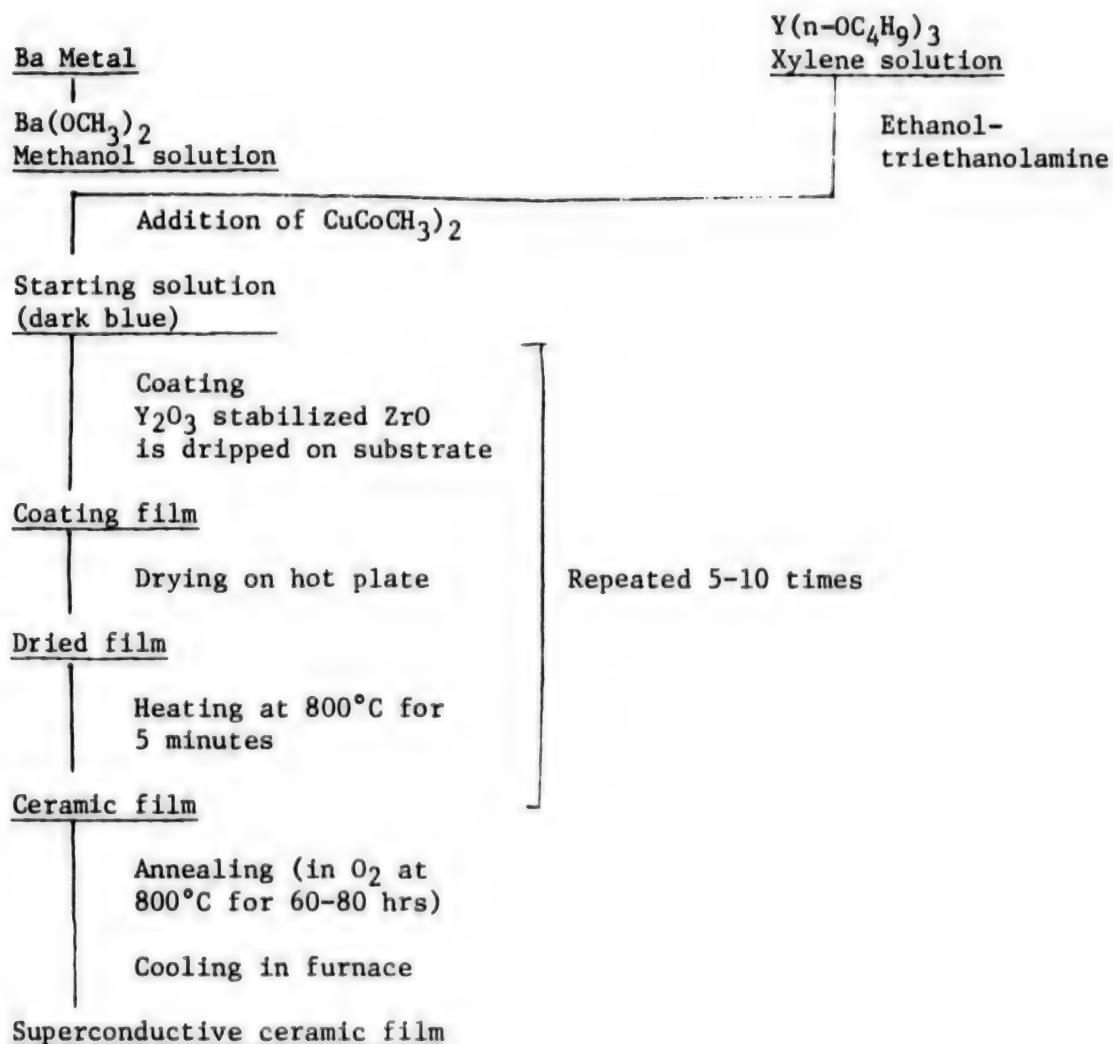


Figure 5. Manufacture of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  High-Temperature Superconductive Film Using Metallic Alkoxide

Under the sol-gel process, the viscosity of a solution achieved through reaction does not necessarily ensure that the solution can be spun. For the solution to be spun, it is necessary that it have a threading property, that its gelation be advanced and that it solidifies in air relatively rapidly when the yarn is drawn.<sup>1</sup> Since the metallic alkoxide solution used for making a coating film, which was referred to in the preceding paragraph, does not display a threading property in a viscose state, it cannot be used for spinning.

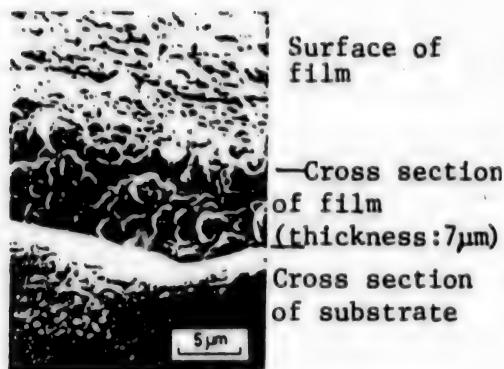


Figure 6. Photo of Cross Section of Coating Film Taken by Scanning Electron Microscope (SEM)

It was heated for 60 hours at 800°C in oxygen atmosphere after releasing coating work 8 times (dripping, drying and heating at 800°C for 5 minutes).

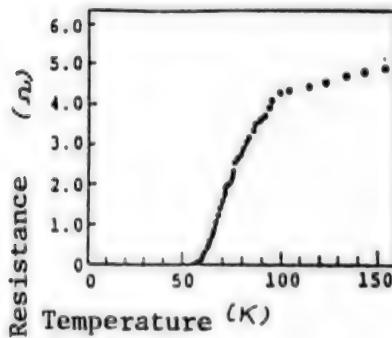


Figure 7. Resistance-Temperature Curve of Coating Film

In the laboratory used by the author's group, fibers were produced from an acetate solution as shown in Figure 2. In the figure, if a transparent blue acetate solution is enriched through reactions to the extent that its volume is reduced to about one-tenth that at the time of preparation, it becomes a transparent and viscose blue gel with a threading property. If a glass rod is lifted from the solution, a fiber more than 1 m long can be drawn. The gel fiber thus spun has a smooth surface, as shown in Figure 8. If it is heated to 900-950°C, a fiber consisting of a  $\text{YBa}_2\text{Cu}_3\text{O}_7-\sigma$  superconductive crystal can be obtained as shown in Figure 9. From such fibers, it is possible to produce a superconductive fiber with a diameter of nearly 1 mm with a  $T_c$ (end) of nearly 80 K, as shown in Figure 10.

Superconductive fibers are produced in this way. If the surface of a fiber is observed by a scanning electron microscope (SEM), crystalline particles can be clearly seen. It has also been found that the surface is quite rough, as shown in Figure 11. Since these fibers are hollow, they are brittle and not flexible. Therefore, it will be necessary to remedy this defect and to produce tough, flexible fibers.

It is thought that the uneven surface and the hollowness are due to an incomplete metal-oxygen-metal bonding (as shown by a red spectrum) in the gel before heating. Such metals as Ba, Y and Cu were linked together through an acetic acid anion and a Cu acetate of Cu dissolved in carbonic acid gas and water gasified at 200-300°C. These elements are apt to escape rapidly.

Figure 12 shows the results of differential thermal analysis and thermogravimetric analysis. The decrease in gravity can be divided into three stages by thermogravimetric curves: under 200°C, from 200-300°C and from 300-400°C. At temperatures below 200°C the water of a solvent evaporated, at 200-300°C gas from the dissolved Cu acetate was discharged, and at 300-400°C gas from the dissolved Ba and Y acetates was discharged, resulting in a decrease of gravity. Table 3 shows collectively the accompanying substances produced during such changes. It was found that Cu, Cu<sub>2</sub>O, Ba(OH)<sub>2</sub> and BaCO<sub>3</sub> were created, that metal-oxygen-metal bonding first occurred at temperatures greater than 400°C, and that a YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\sigma$</sub>  superconductor was produced above 750°C.

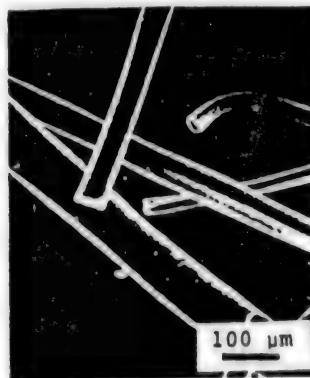


Figure 8. SEM Picture of Gel Fibers

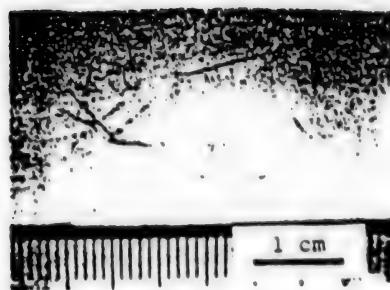


Figure 9. Photo of Calcined Fibers

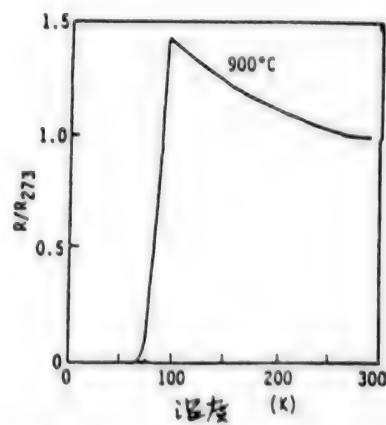


Figure 10. Resistance-Temperature Curve of Calcined Fiber



Figure 11. SEM Picture of Calcined Fiber

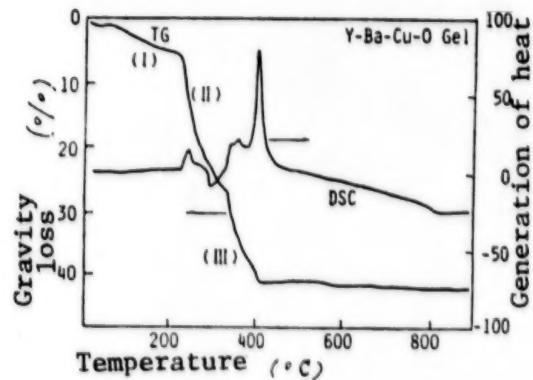


Figure 12. Thermogravimetric and Differential Thermal Analyses by Scanning of Acetate Gel

(1) Desorption of  $\text{H}_2\text{O}$  (2) Disposal of Cu acetate into Cu metal  
(3) Creation of  $\text{Ba}(\text{OH})_2$  and  $\text{BaCO}_3$  from Ba acetate. Change of Y acetate to  $\text{Y}_2\text{O}_3$ .

Table 3. X-Ray Diffraction (Fixation of Phase) of Products When Heating Gel Produced From Acetate at 5°C/min

Temperature (°C)	Phase
90	noncrystalline
230	Cu, Cu <sub>2</sub> O
340	Cu, Cu <sub>2</sub> O, Ba(OH) <sub>2</sub>
450	CuO, BaCO <sub>3</sub> , Y <sub>2</sub> O <sub>3</sub>
600	CuO, BaCO <sub>3</sub> , Y <sub>2</sub> O <sub>3</sub>
750	CuO, BaCO <sub>3</sub> , Y <sub>2</sub> O <sub>3</sub> YBa <sub>2</sub> Cu <sub>3</sub> O <sub>7-<math>\delta</math></sub>
910	YBa <sub>2</sub> Cu <sub>3</sub> O <sub>7-<math>\delta</math></sub>

In view of the changes that occur during heating, in order to produce excellent superconductive fibers it would be good to have a process where the acetates are gradually dissolved under controlled conditions. Additionally, it is necessary to seek a compound other than an acetate whose solution has a threading property and that develops a metal-oxygen-metal bond easily at the time of gelling.

#### 6. Closing Remarks

As discussed above, coating films and fibers of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub>  superconductive ceramics can be manufactured. However, it is necessary to improve their properties and performance further. With regard to fibers in particular, it has only been shown that the sol-gel process can produce fibers. Since superconductive ceramics contain many varieties of ingredients, including three positive elements, it is difficult to discover solvents for producing homogeneous starting solutions by dissolving raw compounds of all ingredients. Therefore, application of the sol-gel process to manufacturing involves several difficulties. However, it is expected that this process will make a marked advance in the future, particularly in view of the fact that it can produce fibers.

### References

1. S. Sakka, *Science of Sol-Gel Process*, Agne Shofu Sha (1988).
2. S. Sakka, "Chemistry of Oxide Superconductor," *Chemistry of Oxide Superconductor*, edited by K. Fueki and K. Kitazawa, Kodansha, pp 167-171 (1988).
3. S. Sakka, H. Kozuka and T. Umeda, *Sol-Gel Processing Applied to Preparation of High-Temperature Superconducting Ceramics*, MRS International Meeting on Advanced Materials, Tokyo, May 30-June 3 (1988).
4. T. Monde, H. Kozuka and S. Sakka, *Chem Lett.*, pp 287-290 (1988).
5. S. Sakka, H. Kozuka and T. Umeda, *Bulletin of Scientific Essays issued by the Japan Ceramics Society*, 96, 448-470 (1988).

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